WL-TR-94-4111

GRIT-BLAST/SILANE (GBS) ALUMINUM SURFACE PREPARATION FOR STRUCTURAL ADHESIVE BONDING

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APRIL 2003

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REPORT DOCUMENTATION PAGE

Form Approved OMB No. 0704-0188

1. REPORT DATE *(DD-MM-YYYY)* 30-04-2003	**2. REPORT TYPE** INTERIM	**3. DATES COVERED** *(From - To)* 1 JUN 92 – 31 AUG 93

4. TITLE AND SUBTITLE
GRIT-BLAST/SILANE (GBS) ALUMINUM SURFACE PREPARATION FOR STRUCTURAL ADHESIVE BONDING

5a. CONTRACT NUMBER F33615-89-C-5643
5b. GRANT NUMBER
5c. PROGRAM ELEMENT NUMBER 62102F

6. AUTHOR(S)
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5d. PROJECT NUMBER 2418
5e. TASK NUMBER 04
5f. WORK UNIT NUMBER 69

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
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8. PERFORMING ORGANIZATION REPORT NUMBER
UDR-TR-94-153

9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES)
MATERIALS AND MANUFACTURING DIRECTORATE
AIR FORCE RESEARCH LABORATORY
AIR FORCE MATERIEL COMMAND
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10. SPONSOR/MONITOR'S ACRONYM(S)
AFRL/MLSA

11. SPONSOR/MONITOR'S REPORT NUMBER(S)
WL-TR-94-4111

12. DISTRIBUTION / AVAILABILITY STATEMENT

APPROVED FOR PUBLIC RELEASE; DISTRIBUTION IS UNLIMITED

13. SUPPLEMENTARY NOTES
THE BULK OF THE WORK REPORTED HEREIN WAS CONDUCTED UNDER THE ABOVE CONTRACT NUMBER DURING THE PERIOD NOTED. ADDITIONAL WORK WAS COMPLETED FROM 1998 TO 2003 AS NOTED IN THE PREFACE.

14. ABSTRACT
A silane surface preparation for on-aircraft adhesive bonding of aluminum, known as the grit-blast/silane (GBS) process, has been optimized. It consists of abrasion using 3M Company Scotch-Brite® pads, aluminum oxide grit blast, and application of an epoxy-functional organosilane coupling agent hydrolyzed in water. The surface preparation has advantages for on-aircraft repair since it does not require the use of acids. The Aeronautical Research Laboratory of the Australian Defence Science Technology Organisation (DSTO) developed a similar procedure that has been used by the Royal Australian Air Force (RAAF) for numerous repairs. Several organizations outside of DSTO have found it difficult to reproduce the good results achieved by DSTO. This investigation was intended to duplicate the DSTO results and discover the reason for discrepancies noted by others. The critical process variables were identified, and the process was optimized. Key process variables investigated include: silane drying temperature and time; silane solution concentration and pH; solution hydrolysis time; silane application time; aluminum oxide grit size; aluminum alloy; silane drying technique; and various time lags between process steps. Surface preparation performance was characterized by means of the ASTM D 3762 wedge test conducted at 120°F and 95-100% relative humidity. Most tests were performed on 2024-T3 bare aluminum. After silane treatment, most test specimens were primed with Cytec Engineered Material's BR 127 and bonded with Cytec FM 73 epoxy film adhesive. After optimization of the process, favorable comparison with phosphoric acid anodization (PAA) was achieved. Several organizations have used the optimized GBS process to apply bonded doublers on aircraft and to treat aluminum for adhesive bonding at the depot level. Detailed GBS procedures are included in the report.

15. SUBJECT TERMS
SILANE COUPLING AGENT ADHESIVE BONDING WEDGE TEST GRIT BLAST ALUMINUM SURFACE PREPARATION

16. SECURITY CLASSIFICATION OF: a. REPORT	b. ABSTRACT	c. THIS PAGE	17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
UNCLASSIFIED	UNCLASSIFIED	UNCLASSIFIED	SAR	71	JAMES J. MAZZA
					19b. TELEPHONE NUMBER *(include area code)* 937-255-7778

Standard Form 298 (Rev. 8-98)
Prescribed by ANSI Std. 239.18

PREFACE

This report primarily documents work conducted by the University of Dayton Research Institute (UDRI) for the U.S. Air Force Research Laboratory Materials and Manufacturing Directorate (AFRL/MLSA)* during the period from June 1992 through August 1993. The effort was conducted by the Plastics, Adhesives, and Composites Laboratory of the UDRI Materials Engineering Division under Air Force contract F33615-89-C-5643. The UDRI Principal Investigator was Ron Kuhbander, and the majority of the laboratory work was conducted by John Stalter. The work was undertaken in order to validate an aluminum prebond surface preparation using a silane coupling agent that was developed by the Air Vehicles Division of the Australian Defence Science and Technology Organisation (DSTO)† and implemented by the Royal Australian Air Force (RAAF) for repair bonding. The intent was to rebaseline the procedure and optimize it by investigating process variables as well as define tolerances for the key steps.

Although the bulk of this report was completed and the report number was obtained soon after the initial UDRI work was finished, it was not published at that time for a variety of reasons. The majority of the data were reported in a paper presented in May 1993‡. The U.S. Air Force implemented grit-blast/silane (GBS) for bonded repairs in August of that year at Warner Robins Air Logistics Center (WR-ALC) where it has been in continual use ever since. During the period 1998 through 2003, sporadic work was conducted to expand the GBS database and better define key variables, particularly optimal silane drying temperature. Significant work was conducted by 1 Lt Jason Avram (AFRL/MLSA) at Wright-Patterson AFB OH, supported by Dan McCray and Jeff Smith of UDRI under Air Force contracts F33615-95-D-5617 and F33615-00-D-5600. In addition, a final silane drying temperature study was undertaken in 2003 by UDRI (McCray and Smith) under Air Force contract F33615-00-D-5600 using panels fabricated by Harold Banks of WR-ALC/EN. Inconsistencies encountered between the silane drying temperature studies are discussed in this report and contributed to its delayed release.

All of the UDRI work for the three contracts was administered by the Principal Investigator, D. Robert Askins. Jim Mazza of AFRL/MLSA was the Air Force Project Engineer for the effort.

This report was prematurely referenced in open literature with a September 1999 date.

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* Air Force Wright Laboratory Materials Directorate (WL/MLSE) in 1993

† DSTO's Aeronautical Research Laboratory (ARL) in 1993; later the Aeronautical and Maritime Research Laboratory (DSTO)

‡ R.J. Kuhbander and J.J. Mazza, 38th International SAMPE Symposium Proceedings, May 1993, p. 1225.

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1 INTRODUCTION

1.1 Surface Preparation

Adherend surface preparation is essential for the successful implementation of adhesive bonding technology. Both initial bond strength and subsequent long-term durability are critically dependent on the interaction between the polymer (adhesive or primer) and a pretreated adherend surface. For metals, surface preparation involves both the removal of weak boundary layers or layers that are chemically incompatible with the adhesive and the formation of stable, adherent layers that are mechanically and chemically compatible with the adhesive[1].

For metal bonding, particularly aluminum, resisting the attack of moisture is the key to long-term durability. Eventually, moisture will gain access to the interface region between the polymer and metal where it can destroy bonds based on weak chemical or physical attractions. It can also cause unstable oxide layers to further hydrate, thereby destroying the metal surface to which the polymer is bound. To achieve good initial adhesion and good moisture durability, contamination and weak native oxide layers must be removed from the metal surface which then must be rendered chemically compatible with the adhesive (or primer). Adhesion and durability are improved if the surface is roughened, particularly at the microscopic level. To achieve long-term moisture durability, a stable oxide layer must be created and moisture ingress must be minimized.

There are many qualified surface preparations for aluminum adhesive bonding. The best treatments yield excellent long-term moisture durability but tend to involve strong acids and other hazardous chemicals that render them unsuitable for some applications, particularly on-aircraft repair. Many approved processes, especially those for repair, do not deliver good durability or even acceptable initial adhesion. In a production or depot setting, facilities are generally available for tankline (dip-tank) processes. These include anodization and etching procedures using strong acids or bases. For aluminum, phosphoric acid anodize (PAA) is the premier treatment, offering good adhesion and durability performance in service. This electrolytic process creates a controlled, microporous oxide surface that is relatively stable. Other acid anodization processes available for aluminum treatment include chromic, sulfuric/boric, and thin sulfuric. Acid etches, such as the various sulfuric acid-sodium dichromate processes, create morphologies on aluminum surfaces without the use of electrical current. However, the structures obtained are generally not as robust as those achieved by anodization and do not provide the same level of interlocking for polymers within the pore structure. For optimal durability, the anodization and etching processes are followed by

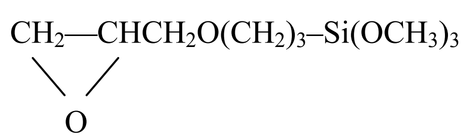
application of a corrosion-inhibiting adhesive primer (CIAP). The primer helps minimize moisture access to the oxide layer and can penetrate micropores to “wet” the surface.

In a repair environment, particularly on-component, surface preparations that provide acceptable bond performance are typically inconvenient or impossible to use. Adaptations of anodize and etch processes have been made, however, these are rarely conducted within optimal processing parameters and are much more difficult to apply than the dip-tank versions. The hazardous materials (strong acids and chromates) are harder to control and can be detrimental to the surrounding structure and personnel. Concerns include corrosion of aluminum due to entrapped acid and embrittlement of high-strength steel. To avoid these problems, simple clean and abrasion processes are often employed, with a significant reduction in performance, particularly moisture resistance.

The grit-blast/silane (GBS) aluminum surface preparation investigated and reported herein does not utilize strong acids. The results have shown that, following the proper procedures, durable bonds similar to those obtained using the state-of-the-art phosphoric acid anodization (PAA) procedure can be achieved. In this study, specimens prepared using both PAA and GBS were primed with a chromated adhesive primer prior to bonding.

1.2 Silane Coupling Agents

Silane coupling agents are a large family of organosilicon monomers with the chemical formula R-SiX_3. In this formula, R is an organic functional group attached to the silicon atom. Most commonly, R consists of a reactive group, R', and a hydrocarbon chain that separates this group from the Si atom. Hydrolyzable groups, X, are converted to silanol groups (SiOH) via hydrolysis[2]. The coupling agent is usually hydrolyzed in an aqueous solution to form the trisilanol (R-Si-$(OH)_3$). The coupling agent used in this study was the epoxy-functional (γ-glycidoxypropyl)trimethoxysilane (γ-GPS). The hydrolyzable groups of this silane are methoxies and the organic reactive group is an epoxy which is separated from the silicon atom by a propylene group:



The function of the coupling agent is to provide stable bonds between two materials that may otherwise not be bondable, such as aluminum oxide and epoxy resin. The improved adhesion provided by silane coupling agents is even more pronounced after moisture exposure[2]. The

mechanism by which the coupling agent improves the hydrothermal stability of interfaces between organic polymers and inorganic substrates is not fully understood. Several theories have been suggested, the most common being the "chemical bonding" theory[3]: the two different types of reactive groups present in coupling agents allow them to chemically bond to both organic polymer and inorganic surfaces. For γ-GPS, the silanol groups bond to the hydroxyl (-OH) groups on the aluminum surface in a condensation reaction, and the epoxy functionality bonds with the amine curative in the adhesive or primer.

Silane coupling agents were developed in the 1940s to pretreat glass fibers for use in composites in order to improve their water resistance[4]. Scientific interest in silane coupling agents has since proliferated for a wide variety of applications, and a large volume of related information can be found in the open literature. The use of silane coupling agents as constituents in aluminum prebond surface treatments is not new; many organizations use or have used silanes in this manner. In the early 1990s, there was increased interest in silanes for prebond treatment of aluminum, primarily because the Aeronautical Research Laboratory (ARL) of the Australian Defence Science and Technology Organisation (DSTO) together with the Royal Australian Air Force (RAAF) had employed silanes in their efforts to repair/reinforce metallic aircraft structure with boron/epoxy patches. Since the RAAF had numerous applications with many years of success, the DSTO boron/epoxy patching process, including silane surface preparation, became increasingly visible as both military and commercial aircraft users around the world were forced to deal with fatigue cracks and other problems associated with their aging aircraft.

Several organizations tried to duplicate the ASTM D 3762[5] wedge test results cited by DSTO for their silane surface preparation. The procedures used by these organizations were gleaned from the open literature and conversations with DSTO. Also, in at least one instance, round robin-type testing was conducted with DSTO[6]. Consistent results duplicating DSTO's data were not obtained. Obviously, some difference in the silane procedure or testing method existed.

1.3 Renewed U.S. Air Force Bonded Repair Interest

Many aircraft in the U.S. Air Force inventory have been in service for relatively long periods. Extended service such as this often leads to the development of fatigue cracks in the aircraft structure. Repair of this type of damage can pose a significant problem. In the past, repairs were usually accomplished by "stop-drilling" the end of the crack followed by application of a mechanically fastened metal patch. Frequently, the original crack would continue to grow or new cracks would initiate at the edges of the patch or at the fastener holes. Bonded patches have advantages over this repair approach in respect to repair flexibility, structural efficiency, and

damage tolerance. For these reasons, the U.S. Air Force began to focus on bonded repairs for metallic structure.

In 1992, a program was initiated by the U.S. Air Force Research Laboratory Materials and Manufacturing Directorate (AFRL/MLSA) to address several technical issues regarding bonded patches: on-aircraft prebond metal surface preparation, adhesive primers, adhesive cure techniques, and nondestructive evaluation. The multitask program was entirely funded by the Air Force Productivity, Reliability, Availability and Maintainability (PRAM) Program Office under PRAM Project No. 34691-01. The second task of the program generated the majority of the GBS data contained in this report. This effort was devised to duplicate the DSTO results and discover the reasons for data discrepancies noted in previous efforts to verify their work. Critical variables were identified and the process was optimized. Although the optimized process is based on the DSTO silane surface preparation, the GBS work presented herein went back to basics, so differences likely exist between the DSTO approach and the AFRL/MLSA GBS procedure. Although both processes use γ-GPS, the particular silane used in this study was Z-6040 from Dow Corning, whereas DSTO originally used Union Carbide's A-187.

2 SURFACE PREPARATION AND TEST PLAN PARAMETERS

The general outline for the DSTO silane surface preparation[7,8] is as follows:

- Degrease with methyl ethyl ketone (MEK)
- Abrade with 3M Company Scotch-Brite® abrasive pads (wet with MEK)
- Clean surface with MEK and unpigmented/unscented tissues until no contamination is visible on fresh tissues
- Grit blast with 45-50 micron aluminum oxide to a fine matte finish (use nitrogen)
- Blow off excess grit with nitrogen
- Wet surface for 10 minutes with a 1% silane solution in deionized or distilled water which has been hydrolyzed (continuous stirring) for 1 hour
- Air dry at ambient temperature or heat gun dry
- Apply adhesive primer

The early DSTO silane work was unprimed, whereas later work included the priming step. The primer is believed to have been BR 127 from Cytec Engineered Materials, Inc. (formerly Cyanamid), since it was used by DSTO in similar work on an anodize surface preparation[9]. DSTO reported durability approaching that of acid anodization when using their silane plus primer treatment, based on wedge test results[10].

No details other than the general outline above were available for the majority of this study. Critical variables, techniques for controlling the critical variables, and tolerances had to be developed. Process variables that were considered included:

(a) silane drying temperature and time
(b) silane solution concentration
(c) hydrolysis time
(d) silane application time
(e) aluminum oxide grit size
(f) silane source
(g) aluminum alloy
(h) pH of silane solution
(i) silane drying technique

The performance of the surface treatment was characterized by means of wedge tests per ASTM D 3762[5]. Additional data in Appendix A include some lap shear and peel test results.

2.1 The Wedge Test

Work in the 1970s conducted by Boeing using double cantilever beam (DCB) specimens demonstrated that environmental exposure of Mode I loaded specimens duplicate the characteristics of in-service disbonds much better than do lap shear or peel tests. Boeing developed the wedge test as a simplified, qualitative version of the DCB specimen for use in controlling factory dip-tank surface preparation processes. The test was also used by Boeing to study operating parameters for the sulfuric acid-sodium dichromate process, leading to its optimization. Later, the wedge test played a critical role in the development of the PAA surface preparation[11]. Early U.S. Air Force work determined the wedge test was able to differentiate between and provide a relative ranking for many of the various parameters that affect adhesive bond joint durability[12]. For these reasons, silane surface preparation variables in this study were evaluated using the wedge test. Although the wedge test, if used correctly, is a good screening tool for surface preparation performance, "qualification" of a surface preparation for application ultimately requires additional testing.

The wedge test is described in ASTM D 3762[5]. For the test, five 1in x 6in test specimens are machined from a panel fabricated by adhesively bonding two 6in x 6in x 0.125in surface-treated adherends. A 0.125 inch-thick wedge is then driven into the adhesive bondline between the two adherends of each specimen. Cracks created by driving the wedge must be within the adhesive layer. The lengths of these initial cracks depend on the adhesive and are typically in the range of 1-1.5 inches for common 250°F-curing modified epoxies. The specimens are placed in a conditioning environment, usually hot/wet at 120°F or 140°F & 95%-100% relative humidity (RH). Initial crack lengths are recorded prior to conditioning, and crack extension during the test is recorded periodically. Ideally, crack growth during hot/wet conditioning will remain within the adhesive layer. This indicates the interface between the polymer and metal is more durable (under the given conditions) than the adhesive. The moisture durability of the interface is the weak link for in-service bonds and is directly dependent on the metal surface preparation[13].

The ASTM D 3762 standard is not particularly useful beyond defining the test specimen configuration and general test protocol. It only suggests possible hot/wet conditioning parameters, test duration, and pass/fail criteria. ASTM D 3762 (or any standard) cannot set explicit conditioning and pass/fail criteria. Any such determination is subjective since there is no definitive way to quantify the moisture durability of a bonded joint in a manner that can be correlated with service life. Therefore, they are set by individual organizations as seen fit.

AFRL/MLSA considers the wedge test to be “semiquantitative,” with the true pass/fail criterion being the failure mode that is determined upon opening the bonded specimens after conditioning. Surface preparation performance is measured against that of a baseline process, such as PAA/BR 127, that has proven service reliability and much additional qualification testing. Initial crack length serves as an indicator of test severity (of the stress induced at the crack tip) to ensure proper comparison with the baseline process. Crack extension in the hot/wet environment is of less importance but can be used to predict the failure modes of yet unopened specimens (e.g. sudden large crack extension is indicative of an interfacial failure). It is imperative that all wedge test fabrication and testing variables, other than the metal surface preparation, be the same in order to obtain comparisons between processes.

The preferred AFRL/MLSA wedge test conditions are 140°F & 95-100% RH. These conditions were part of the test matrix used in the Air Force’s Primary Adhesively Bonded Structure Technology (PABST) program. This program was a major undertaking that defined the state-of-the-art for structural adhesive bonding. It also identified PAA/BR 127 (with Cytec FM 73M adhesive) as a reliable system for bonding primary aircraft structure[14,15]. The AFRL/MLSA test duration is 28 days, with crack extension recorded at several intervals during the hot/wet conditioning. Pass/fail is based on the failure mode of the opened specimen after 28 days. Passing requires a 95% or greater “cohesive” failure mode (crack remaining within the adhesive layer). Interfacial failures or failures within other layers in the bondline, such as the primer, are not considered acceptable. For a passing specimen, any so-called “adhesive” failure mode (interfacial, typically at the metal surface) must comprise 5% or less of the specimen test area and must be confined to the specimen edges. Typically, crack lengths are reported as the average of the five specimens in a panel. Often, panel failure modes are given as a “percent cohesive” that is the average for the 5 specimens. This can lead to confusing results, especially when the percent cohesive failure varies considerably between specimens in the panel. In this study, failure modes for individual specimens were not averaged. For a panel to “pass,” all five specimens had to exhibit cohesive failure (95% or greater). Panels with specimens yielding both “adhesive” and “cohesive” failures were said to have a “mixed” failure mode and were not considered “passing.”

The wedge test is a contrived test that attempts to simulate the long-term effects of a moisture environment in a short period. It does not generate design data. Adhesives in service are not subjected to the types of loads and conditions seen in the wedge test. It is likely the test is too stringent. Surface preparations that do not meet the AFRL/MLSA “pass” criterion may perform for long periods in service. Without a quantitative correlation between the wedge test and

service life, AFRL/MLSA has adopted the conservative approach of requiring surface preparations to yield 120°F wedge test results comparable to those of PAA/BR 127 which does meet the criterion. Other organizations have adopted different requirements.

Some organizations have compared wedge test performance with service life. AFRL/MLSA considers these correlations specific to the adhesive systems/applications evaluated and not transferable to dissimilar situations. Boeing wedge test pioneers considered the test to be "qualitative and empirical," so they looked to correlations with service history to establish pass/fail criterion. This led to the "arbitrary" pass/fail criterion adopted for the process control wedge test: for ten specimens, no individual crack extension may exceed 0.75in after 60-75 minutes exposure to 120°F and 100% RH[16]. Although the test has apparently performed well in its process control role, surface preparations can meet the original Boeing criterion while exhibiting complete interfacial failures. This type of performance is considered unacceptable today by the majority of the aerospace adhesive bonding community. The community has a great need for better-defined surface preparation qualification approaches, including standardized wedge test criterion, and more/better correlations between in-service surface preparation performance and the wedge (or other) test.

2.2 Initial Silane Surface Preparation Evaluations

Wedge test panels were prepared using 2024-T3 bare aluminum with both PAA (ASTM D 3933) and DSTO silane surface preparations (best attempt based on literature data). These were primed with BR 127 and bonded with FM 73 adhesive. Primer and adhesive were cured in accordance with the manufacturer's recommendations. BR 127 primer was dried at ambient conditions (≈70°F) for 30 minutes followed by 1 hour at 250°F. FM 73 adhesive (0.06 psf) was cured for 1 hour at 250°F under 35-40psi pressure after a heat-up at approximately 5°F/minute.

The wedge test specimens were machined, wedges were inserted, and initial crack lengths were recorded per the ASTM standard. The specimens were then exposed to 140°F & 95-100% RH for 28 days and crack extension was periodically measured. After the 28-day exposure, failure modes for the PAA-prepared specimens were cohesive within the FM 73 layer. The failure modes for silane-prepared specimens changed from cohesive to adhesive after 24 hours of exposure. These interfacial failures typically extended across the full width of the specimen. In addition, crack extension was significantly higher for the silane-treated specimens than for their PAA counterparts. Since it was not clear if the results reported by DSTO were obtained for panels prepared using the adhesive and primer conditions shown above, several wedge test panel

bonding parameters were investigated: pressure application (vacuum versus press); bondline thickness (one adhesive ply versus two); primed versus unprimed; and adhesive cure temperature. None of the silane-treated panels yielded specimens with cohesive failures or crack extensions similar to those of PAA-treated specimens.

An additional literature search was conducted to determine some important parameter or step that might have been overlooked, thereby causing the poor performance. The search revealed other organizations evaluating DSTO's silane surface treatment saw similar results[17,18]. Importantly, the literature showed DSTO wedge test aging was conducted at 122°F (50°C) rather than 140°F for various times, usually 28 days or longer. DSTO pass/fail criteria for the wedge test, necessary for data comparisons, were not clear. Crack growth and fracture energy were definitely important[19]. Failure mode was also a consideration, with 90% cohesive failure apparently indicating good performance[20]. Taken with previous literature, the new search also revealed a wide range of silane drying conditions for the DSTO process: from ambient temperature to 80°C (176°F) to 110°C (230°F) to unknown (via heat gun). Use of elevated-temperature drying appeared to be directed at moisture removal from the adherend surface to reduce porosity in the adhesive bondline as opposed to being a requirement for the silane treatment itself[21,22,23,24].

An additional wedge test panel was fabricated and specimens were subjected to 120°F & 95-100% RH conditions. Silane drying was still conducted at ambient laboratory temperature (70°F). Test results are shown in Table 2.2-1. It is apparent that reducing the conditioning temperature significantly improved the failure mode and decreased crack growth. Since the objective of the work was to duplicate the DSTO results and determine tolerances for the important process variables, work continued using the wedge test at 120°F & 95-100% RH, and silane drying temperature was investigated as a key variable.

Table 2.2-1 Effect of Conditioning Temperature on Wedge Test Performance

Temp. (°F)	Initial (in)	Cumulative Crack Growth (in)				Total (in)
		1 hr	4 hr	8 hr	24 hr	
140	1.20	0.14	0.31	0.41	0.58	1.78*
120	1.18	0.09	0.13	0.23	0.35	1.53†

*: complete interfacial (metal-to-primer) failures across the specimens' widths
†: mostly cohesive failures within the adhesive layer with interfacial failure "spotted" across the test area

3 EFFECT OF PROCESS VARIABLES ON GRIT-BLAST/SILANE SURFACE PREPARATION

Several baseline wedge test panels were fabricated using one ply of 0.06 psf areal weight FM 73 adhesive with BR 127 primer, both cured per the manufacturer's recommendations. Specimens were conditioned at 120°F & 95-100% RH, and crack growth was recorded. None yielded results comparable to PAA, and all of the failure modes were adhesive (at the metal interface) after several hours in the humidity environment.

Baseline wedge test specimens were fabricated by first MEK-wiping aluminum adherends with an unscented/unpigmented tissues to degrease the surface. They were then deoxidized using 3M Company Scotch-Brite (7447B – Type A very fine) until the bonding surface had a "shiny" appearance. Adherends were again wiped with MEK to remove residue left from the deoxidation process. The adherends were grit-blasted with 50-micron aluminum oxide to a uniform, fine matte finish. Dry, oil-free nitrogen was used for the grit blast process. Excess aluminum oxide grit was blown off using nitrogen. The bonding surface of each adherend was then completely covered in silane for 10 minutes. The silane solution used was a 1% Dow Corning Z-6040 (γ-GPS) in deionized water that was hydrolyzed for one hour before use. After the 10-minute silane application (in the horizontal position), each adherend was grasped at the edges, stood upright (vertical), and the excess solution was removed from the adherend surface by touching the lower edge onto an absorbent wipe. The adherends were then air dried at ambient temperature until water was not apparent on the surface. BR 127 adhesive primer was applied, adherends were bonded, and panels were machined to obtain the wedge test specimens.

The grit-blast spray gun used for the initial work was a S.S. White, Model F. This spray gun presented a problem because it blasted a very small area, making it difficult to slightly overlap each succeeding pass and obtain a uniform surface. At the time, it was believed this may be the source of the poor wedge test performance. Another spray gun was purchased: Speedaire portable sandblaster No. 6W614 with a 0.20in diameter tungsten carbide nozzle. This gun, angled at about 45° to the panel surface, sprayed a larger area, making it much easier to slightly overlap each succeeding pass. This resulted in a very uniform, fine matte or frosty-appearing finish. However, wedge test specimens prepared after grit-blasting with this gun exhibited crack extensions similar to those previously reported and yielded adhesive failure modes.

3.1 Effect of Silane Drying Temperature

Since the extended literature search revealed a wide variety of reported silane drying temperatures, this variable was the first evaluated. In this effort, all variables were held fixed (as best as possible) other than the one being evaluated. Interactions between variables were not investigated, despite the fact some are of obvious importance, such as silane drying temperature interaction with silane drying time.

3.1.1 Initial Silane Drying Temperature Evaluation (1992-93)

Wedge test panels were prepared as before, but the silane solution was dried using a heat gun after the 10-minute application time. Drying temperature and uniformity across the panel were not determined. The failure mode was interfacial at the metal-primer interface although crack growth observed in the specimens was slightly less than that seen for specimens that were air dried at ambient conditions. Panels were then prepared with controlled silane drying temperatures of 140°F and 175°F. The silane-treated adherends were inserted upright (vertical) in a rack and immediately placed in a preheated oven at the selected drying temperature for 1 hour. Again, the resulting wedge test specimens were exposed to the hot/wet environment. There was a noticeable change in failure mode accompanied by a reduction in crack growth rate during the test. As the drying temperature increased, so did the percentage of cohesive failure. Additional panels were then fabricated with controlled drying temperatures up to 225°F. The results, shown in Table 3.1-1, clearly indicate that the 200°F drying temperature yielded the best results. Close examination of the failure modes showed the specimens dried at 200°F exhibited 100% cohesive failures, similar to those prepared by PAA. The smallest crack growths in the study were also seen for those panels dried at 200°F for 1 hour. The results shown are an average of at least five specimens (one panel) and in most cases, including 200°F, are the average of 20 specimens (four panels). Figure 3.1-1 illustrates the failure mode comparison between air dry and drying at 200°F. All 200°F-dried specimens resulted in cohesive failures similar to the one shown in the figure.

Table 3.1-1 Effect of Silane Drying Temperature on Wedge Test Performance (120°F & 95-100% RH)

Drying Temp. and Technique	Initial (in)	Cumulative Crack Growth (in)						Total (in)
		1 hr	4 hr	8 hr	24 hr	7 days	28 days	
1 hr at 72°F	1.20	0.07	0.13	0.19	0.33	0.50	0.59	1.79a
Heat gun dry	1.22	0.06	0.09	0.12	0.21	0.39	0.54	1.74a
1 hr at 140°F	1.20	0.07	0.10	0.14	0.20	0.27	0.40	1.60a
1 hr at 175°F	1.16	0.07	0.09	0.10	0.13	0.23	0.39	1.55m
1 hr at 200°F	1.18	0.05	0.08	0.09	0.11	0.19	0.19	1.37c
1 hr at 210°F	1.21	0.06	0.07	0.09	0.10	0.15	0.25	1.46m
1 hr at 215°F	1.12	0.06	0.10	0.12	0.15	0.26a	---	---
1 hr at 220°F	1.14	0.07	0.09	0.12	0.20	0.36a	---	---
1 hr at 225°F	1.26	0.11	0.22	0.33	0.49a	---	---	---

a: adhesive failure mode at the metal interface with the primer
c: cohesive failure mode within the adhesive layer (95% or greater)
m: mixed failure mode with some specimens exhibiting adhesive failure and others having cohesive failure within the adhesive layer

Figure 3.1-1: Comparison of FM 73 Wedge Test Failure Modes for GBS Processing with Silane Drying at Ambient Conditions and 200°F

3.1.2 Additional Silane Drying Temperature Evaluations (1998-99)

In 1997 and 1998, significant DSTO data pertaining to the cause and effect of void formation during vacuum cures of epoxy adhesives began to appear in the open literature[25,26]. Other work reported by DSTO at that time also made it clear that the 110°C silane drying temperature was becoming routine, although 80°C still appeared in the contemporaneous DSTO literature. In 1998, it was learned that the Royal Australian Air Force (RAAF) routinely dried the silane in their process at temperatures above the maximum found to be acceptable in the 1992-93 UDRI study. Until that time, AFRL/MLSA believed the RAAF employed the 200°F (93°C) drying temperature. Since the RAAF was using the 110°C silane drying temperature and DSTO produced good wedge test results with it, additional drying temperature studies were undertaken by AFRL/MLSA and UDRI. The concern was not for bondline porosity but the discrepancy between the wedge test results from the two organizations. In addition, a higher acceptable silane drying temperature was seen as advantageous since it would expand the optimal range for the process implemented within the U.S. Air Force, making it easier to obtain good results on structures with “heat sinks” that lead to rather large temperature ranges during silane drying.

An attempt to verify the work accomplished in 1992-93 was undertaken during the 1998-99 timeframe. GBS wedge test specimens were manufactured using the same silane drying temperatures and times initially investigated, again with BR 127 primer and FM 73 adhesive. The only difference in the surface preparation process occurred after the silane application step. Subsequent to the 10-minute silane application time, excess silane was blown off the adherend surfaces with clean, dry nitrogen instead of being allowed to drain off and be absorbed by Duralace Aerospace Wipes. This step was changed to produce a process that was amenable to on-aircraft applications. It had been validated via wedge tests conducted by mechanics at WR-ALC as part of their certification process (Appendix B). Data from the additional silane drying temperature wedge tests (performed at 120°F & 95%-100% RH) are given in Table 3.1-2. The results shown are an average of five specimens (one panel) for three of the cases (175°F, 180°F, and 240°F) and an average of 10 specimens (two panels) for the rest. Typical failure modes are shown in Figure 3.1-2.

Table 3.1-2 Effect of Silane Drying Temperature on Wedge Test Performance (120°F & 95-100% RH) (1998-99 Results)

Drying Time and Temperature	Initial (in)	Cumulative Crack Growth (in)					Total (in)
		1 hr	8 hr	24 hr	7 days	28 days	
1 hr at 175°F	1.07	0.08	0.11	0.12	0.14	0.17	1.24c
1 hr at 180°F	1.12	0.08	0.10	0.11	0.22	0.35	1.47a
1 hr at 200°F	1.06	0.08	0.10	0.10	0.13	0.18	1.24a
1 hr at 210°F	1.06	0.06	0.07	0.08	0.09	0.18	1.24c
1 hr at 220°F	1.06	0.05	0.08	0.09	0.10	0.13	1.19c
1 hr at 230°F	1.08	0.06	0.08	0.09	0.14	0.18	1.26c
1 hr at 240°F	1.04	0.10	0.12	0.12	0.17	0.19	1.23a

The 1998-99 data contradict the original UDRI work and concur with the DSTO elevated-temperature silane cure results. The 1992-93 data identified 1 hr at 200°F as the optimal silane drying time and temperature, with silane drying temperatures over 210°F leading to degradation of durability. This necessitated restrictions on the upper drying temperature for the U.S. Air Force mechanics performing the process. The later data indicate that drying silane between 210°F and 230°F for 1 hr are the best conditions, with 220°F as optimal. Furthermore, the adhesive environmental durability properties, based on the wedge test failure mode, do not start dropping off until the drying temperature is over 230°F. The 200°F specimens led to adhesive failures (at the metal interface) exceeding 5% with a small amount found away from the edges. This was completely contrary to the 1992-93 work that consistently resulted in 100% cohesive failures at this condition. Good performance for 175°F (80°C) drying contradicts the results of the earlier UDRI work and supports DSTO use of this temperature, but it does not make sense in light of the poor 180°F-drying results.

Although the 200°F drying temperature resulted in nearly "cohesive" failures, the results were obviously not as good as those produced in 1992-1993, as can be seen by comparing Figure 3.1-1 and Figure 3.1-2. This was particularly disturbing since 200°F was set as the optimal silane drying temperature for a fielded process. In an effort to resolve this discrepancy, additional silane drying studies were conducted on more than one occasion with several individuals fabricating specimens. These results, shown in Appendix C, reveal poor or marginal performance at nearly all temperatures. The cause for this is unknown. Water pH, silane age, and "bad" primer were all investigated as possible contributors. No explanation could be found for this inability to repeat earlier results. Based on the fracture surfaces of the adhesive and the fact positive pressure was used to cure the adhesive, water on the adherend surfaces is not

considered a factor. The results may show the GBS process is less robust than first thought. However, in-service performance has been excellent to date with 200°F as the nominal silane drying temperature.

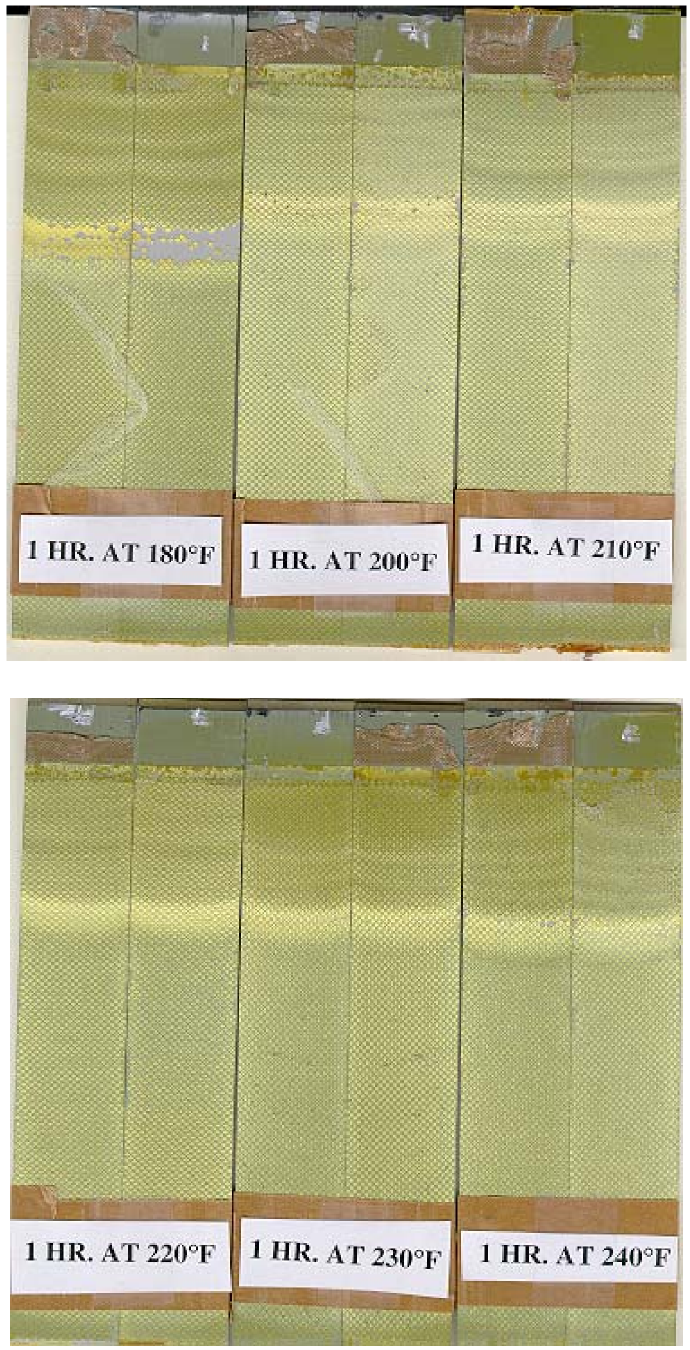


Figure 3.1-2: Comparison of FM 73 Wedge Test Failure Modes for GBS Processing with Silane Drying Temperatures from 180°F to 240°F

The remaining sections this report (other than appendices) refer to 1992-93 work, and the report text was written in 1994. These sections refer to 200°F as the "optimum" drying temperature for silane, and the discussions relating to process variables use this condition. The results obtained for the other process variables should be valid, since 1 hr at 200°F is still considered an acceptable condition for silane drying, despite the Appendix C data.

3.2 Effect of Drying Time at 200°F

Once the "optimum" drying temperature was established as 200°F, the minimum dry time at this temperature was determined. Wedge test panels were prepared using the GBS procedure with various silane drying times at 200°F. The minimum acceptable drying time appears to be 30 minutes. The failure mode is 100% cohesive after drying for 60 and 90 minutes, and cohesive (>95%) for the 30-minute dry time. The lowest total crack length was obtained after 90 minutes. The results for various drying times are shown in Table 3.2-1. The drying times were not evaluated with temperatures other than 200°F, so the interaction between time and temperature was not studied. Unless otherwise noted, the drying time and temperature selected for subsequent work with other process variables was 1 hour at 200°F.

Table 3.2-1 Effect of Silane Drying Time at 200°F on Wedge Test Performance (120°F & 95-100% RH)

Drying (1) Time (min)	Initial (in)	Cumulative Crack Growth (in)						Total (in)
		1 hr	4 hr	8 hr	24 hr	7 days	28 days	
15	1.26	0.08	0.10	0.12	0.16	0.24	0.29	1.55m
30	1.09	0.03	0.07	0.09	0.11	0.19	0.27	1.36c
60	1.18	0.05	0.08	0.09	0.11	0.19	0.19	1.37c
90	1.06	0.05	0.08	0.10	0.11	0.14	0.22	1.28c

3.3 Effect of Percent Silane

The percent silane in water should be 1% according to the DSTO literature[7,8], but the limits are not reported. It is not clear from the literature if other concentrations have been investigated. According to Dow Corning, a very dilute aqueous solution will promote adhesion[2].

In this effort, the percent silane was varied in an attempt to determine the minimum, optimum, and maximum concentrations. After application, the silane was oven dried at 200°F for 1 hour. Concentrations varied from 0.5 to 5%, and the wedge test results are shown in Table 3.3-1. It is

clear that the minimum acceptable is 1% and that 5% is too high. The failure modes for wedge test specimens within the 1% to 3% concentration range were all cohesive. Based on these results, the percent silane concentration should apparently be 1^{-0}_{+2}. As previously discussed, the water used in this study was deionized with a slightly acidic pH. Generally, the aqueous γ-GPS solutions are more soluble and remain stable longer when the pH is lower (approximately 4). This is discussed in detail in Section 3.9.

Table 3.3-1 Effect of Percent Silane on Wedge Test Performance (120°F & 95-100% RH)

Percent Silane	Initial (in)	Cumulative Crack Growth (in)						Total (in)
		1 hr	4 hr	8 hr	24 hr	7 days	28 days	
0.5	1.23	0.07	0.10	0.15	0.18	0.34	0.38	1.61a
0.75	1.22	0.06	0.10	0.12	0.14	0.25	0.34	1.56a
1	1.18	0.05	0.08	0.09	0.11	0.19	0.19	1.37c
2	1.20	0.05	0.08	0.10	0.11	0.15	0.18	1.38c
3	1.22	0.06	0.10	0.11	0.11	0.15	0.17	1.39c
5	1.17	0.09	0.13	0.13	0.17	0.37	0.47	1.64a

3.4 Effect of Hydrolysis Time

Epoxy-functional silanes must be hydrolyzed to form the siloxane functional groups for coupling to aluminum oxide. The minimum time recommended for hydrolysis according to Dow Corning is 15 minutes[2]. One-percent solutions of silane and deionized water were prepared. The hydrolysis time, or that time the solution was allowed to set prior to its application on the aluminum, varied from 0.5 to 24 hours. The solution was continually mixed during this period using a stir bar and magnetic mixer. The wedge test results are shown in Table 3.4-1. Based on the results after 28 days in humidity, the hydrolysis time should be held to about 1 hour. For the times investigated, the only specimens to yield cohesive failure after 28 days were those with 0.75 and 1-hour hydrolysis times. The remaining times investigated , other than 24 hours, yielded some specimens with cohesive failure modes. It must be noted that the specimens for the 0.75-hour hydrolysis time had a significantly larger initial crack length, so the test was not as stringent as for the 1-hour case.

Table 3.4-1 Effect of Hydrolysis Time on Wedge Test Performance (120°F & 95-100% RH)

Hydrolysis Time (hr)	Initial (in)	Cumulative Crack Growth (in)						Total (in)
		1 hr	4 hr	8 hr	24 hr	7 days	28 days	
0.5	1.30	0.08	0.09	0.14	0.21	0.40	0.49	1.79a
0.75	1.26	0.07	0.09	0.11	0.15	0.20	0.21	1.47c
1	1.18	0.05	0.08	0.09	0.11	0.19	0.19	1.37c
4	1.04	0.03	0.06	0.10	0.14	0.35	0.43	1.47m
6	1.09	0.04	0.07	0.10	0.17	0.32	0.39	1.48m
8	1.19	0.08	0.11	0.13	0.16	0.21	0.30	1.49m
15	1.23	0.08	0.10	0.13	0.16	0.29	0.34	1.57m
24	1.27	0.06	0.10	0.14	0.27	0.47	0.56	1.83a

3.5 Effect of Silane Application Time

After the 1% silane solution was hydrolyzed, it was applied to the grit-blasted aluminum surface by brushing from the center toward and past the edges using a natural bristle brush. This aided in removal of residual aluminum oxide grit. Some residual grit remained and was not detrimental. A water-break-free surface was obtained when the silane solution was applied to the grit-blasted surface; when a water-break-free surface was not obtained, the panel was reprocessed (cleaned, abraded, and grit-blasted). The metal surface was kept wet for the intended silane application time by occasionally spreading the solution with the brush. The edges tended to dry first, but continuous application was still accomplished from the center toward the edges. According to the available literature, the application time should be 10 minutes. Theoretically, according to Dow Corning, the time required should be brief. Wedge test panels were prepared varying the application time from 5 to 30 minutes. The results are shown in Table 3.5-1. According to the data, the most durable bonds were obtained using application times from 10 to 20 minutes. These specimens all exhibited cohesive failure after 28 days of humidity exposure.

Table 3.5-1 Effect of Silane Application Time on Wedge Test Performance (120°F & 95-100% RH)

Application Time (min)	Initial (in)	Cumulative Crack Growth (in)						Total (in)
		1 hr	4 hr	8 hr	24 hr	7 days	28 days	
5	1.19	0.08	0.11	0.13	0.18	0.37	0.42	1.61a
10	1.18	0.05	0.08	0.09	0.11	0.19	0.19	1.37c
15	1.07	0.03	0.08	0.09	0.13	0.18	0.24	1.31c
20	1.05	0.04	0.08	0.09	0.10	0.13	0.17	1.22c
25	1.08	0.04	0.09	0.13	0.20	0.37	0.43	1.51a
30	1.21	0.06	0.10	0.10	0.11	0.25	0.28	1.49m

3.6 Effect of Aluminum Oxide Grit Size

According to the literature available, the aluminum surface to be prepared for bonding should be grit-blasted to a fine matte finish with 45-50 micron aluminum oxide[7,8]. In this work, nitrogen was used in the blast system to prevent contamination from water or oil. The aluminum oxide was not recycled, to prevent possible contamination. Wedge test panels were prepared by grit-blasting with 50-, 86-, 142-, and 266-micron aluminum oxide. The test data are shown in Table 3.6-1. Crack growth was monitored for 7 days. The failure modes were mostly cohesive after 7 days in humidity although a slight increase in crack growth was observed when using 86- or 142-micron aluminum oxide compared to the 50-micron. When using 266-micron grit, the failure mode was mostly adhesive and a larger crack length was measured.

Table 3.6-1 Effect of Aluminum Oxide Grit Size on Wedge Test Performance (120°F & 95-100% RH)

Aluminum Oxide (microns)	Initial (in)	Cumulative Crack Growth (in)					Total (in)
		1 hr	4 hr	8 hr	24 hr	7 days	
50	1.18	0.05	0.08	0.09	0.11	0.19	1.37c
86	1.16	0.09	0.12	0.12	0.16	0.25	1.41c
142	1.17	0.05	0.09	0.09	0.14	0.26	1.43c
266	1.12	0.05	0.09	0.11	0.20	0.33	1.45a

3.7 Effect of Silane Source

The particular γ-GPS silane used for this study was Z-6040 from Dow Corning. Another chemically identical silane, A-187 (from Union Carbide in 1993), was used by DSTO. Wedge

test panels were prepared with the A-187 silane and directly compared to results obtained using Z-6040, using the same process conditions. Both silanes were dried at RT (≈70°F), 140°F, 175°F, and 200°F. The results are shown in Table 3.7-1. No appreciable differences could be seen except at 200°F where two A-187 specimens exhibited adhesive failures and increased the average crack length. Actually, the differences between the Z-6040 and A-187 panels at 200°F are minimal and cannot be considered significant based on five specimens from one panel with mixed failure modes. Again, the 175°F silane drying temperature yielded good wedge test results.

Table 3.7-1 Effect of γ-GPS Coupling Agent Source on Wedge Test Performance (120°F & 95-100% RH)

Silane	Drying Temp.	Initial, (in)	Cumulative Crack Growth (in)					Total (in)
			1 hr	4 hr	8 hr	24 hr	7 days	
Z-6040	RT	1.20	0.07	0.13	0.19	0.33	0.50	1.70a
A-187	RT	1.19	0.09	0.13	0.20	0.29	0.40	1.59a
Z-6040	1 hr at 140°F	1.20	0.07	0.10	0.14	0.20	0.27	1.47m
A187	1 hr at 140°F	1.14	0.09	0.11	0.13	0.18	0.27	1.41m
Z-6040	1 hr at 175°F	1.16	0.07	0.09	0.10	0.13	0.23	1.49c
A-187	1 hr at 175°F	1.11	0.08	0.09	0.12	0.17	0.21	1.32c
Z-6040	1 hr at 200°F	1.18	0.05	0.08	0.09	0.11	0.19	1.37c
A-187	1 hr at 200°F	1.16	0.08	0.10	0.11	0.15	0.25	1.41m

3.8 Effect of Aluminum Alloy

The aluminum alloy used in this program was bare 2024-T3 since it is a widely used in the aerospace industry and most of the published work with silane coupling agents on aluminum has been accomplished with this alloy. Other alloys, such as 7075-T6, are also widely used on aircraft. Wedge test panels were prepared with 7075-T6 and compared to 2024-T3. The same process conditions were used for each alloy. The data are shown in Table 3.8-1. After 7 days in humidity, the failure mode remained cohesive, but crack growth rate was slightly higher for the 7075-T6 alloy, despite longer initial cracks. After 28 days in humidity, the failure mode was mixed for 7075-T6 and the crack length was slightly higher. It would not be surprising for 7075-T6 to perform differently than 2024-T3 since the former is harder and may not result in the same surface morphology when grit-blasted. Also, the major alloying elements differ. However, significant wedge test data generated by WR-ALC mechanics show good performance of GBS on 7075-T6 (Appendix B).

Table 3.8-1 Effect of Aluminum Alloy on Wedge Test Performance (120°F & 95-100% RH)

Aluminum Alloy	Initial (in)	Cumulative Crack Growth (in)						Total (in)
		1 hr	4 hr	8 hr	24 hr	7 days	28 days	
2024-T3	1.18	0.05	0.08	0.09	0.11	0.19	0.19	1.37c
7075-T6	1.26	0.07	0.10	0.12	0.16	0.25	0.33	1.59m

3.9 Effect of Water Purity and pH

Epoxy-functional silanes are soluble in water, alcohols, and other solvents[2]. In this investigation, only dilute aqueous solutions were considered. Silane and water are not readily soluble, but using water with a slightly acidic pH aids the process. Dow Corning recommends a pH near 4[2]. A 1% silane/deionized water solution should form a clear homogeneous liquid when hydrolyzed. UDRI in-house deionized water was used throughout this program; its pH was periodically checked and found to be about 5. The higher the silane concentration, the more acidic the water should be to insure complete solubility and stability. The pH of water can be adjusted by adding a slight amount of acetic acid.

Silane solutions were prepared with water having pH values of 4, 5, 6, and 7.2. The pH was adjusted with acetic acid to obtain a more acidic water and with sodium hydroxide to obtain a more basic water. The results are shown in Table 3.9-1. All of the failures were cohesive for panels prepared with a water having acidic pH and adhesive (at the metal-primer interface) for the panel with basic pH (near neutral).

Table 3.9-1 Effect of Water pH on Wedge Test Performance (120°F & 95-100% RH)

Water pH	Initial (in)	Cumulative Crack Growth (in)						Total (in)
		1 hr	4 hr	8 hr	24 hr	7 days	28 days	
4	1.29	0.07	0.09	0.11	0.11	0.17	0.22	1.51c
5	1.18	0.05	0.08	0.09	0.11	0.19	0.19	1.37c
6	1.31	0.07	0.08	0.09	0.10	0.14	0.19	1.50c
7.2	1.24	0.10	0.14	0.18	0.28	0.42	0.56	1.80a

3.10 Miscellaneous Effects

Optimization of the GBS surface preparation was performed in the laboratory under ideal conditions. Field or depot-level repairs are usually performed under less desirable conditions.

For instance, temperature, humidity, and airborne contaminants are less controllable outside of the laboratory or clean-room environment. These factors do not appear to cause any more difficulties for the GBS surface preparation than they do for other processes. However, several issues related to accessibility of the repair area and availability of equipment do influence the GBS process. Many of these involve the time lags between process steps.

3.10.1 Effect of Silane Drying Technique

In the laboratory, it is quite easy to remove the excess silane solution from a treated panel and place it in a preheated oven without a time delay. However, on-aircraft repairs require some time to set up the necessary infrared heat lamps or other means to dry the silane. Panels were prepared using the optimized process conditions developed in the laboratory, except the silane solution was allowed to dry at ambient temperature (≈70°F) prior to oven dry at 200°F to simulate the real-world lag time. Ambient-temperature air dry times of 30 and 60 minutes were evaluated. Wedge test performance for the specimens was significantly inferior to the case of oven dry with no delay. As the silane solution dried under ambient conditions, the panels dried from the corners and edges toward the center. As the silane dried, it left dark colored rings, possibly deposits of residual aluminum oxide. Although this was not investigated, it is suspected that deposits do not affect the bond strength but are merely unsightly. In any event, given the wedge test results for the delayed drying cases, another approach had to be developed. A panel was prepared in which the silane solution force-dried with clean nitrogen after the 10-minute application time. Drying was from the center toward the edges in a circular motion. When the adherend surfaces were nitrogen dried (blown off), they exhibited only slight dark-colored streaks. The panel was then dried for 1 hour at 200°F by placing it in a preheated air-circulating oven. The wedge test data show equivalence with the no-delay (immediate oven dry) case, as shown in Table 3.10-1.

Table 3.10-1 Effect of Silane Drying Technique Prior to Oven Dry for One Hour at 200°F on Wedge Test Performance (120°F & 95-100% RH)

Predrying Condition	Initial (in)	Cumulative Crack Growth (in)						Total (in)
		1 hr	4 hr	8 hr	24 hr	7 days	28 days	
None	1.18	0.05	0.08	0.09	0.11	0.19	0.19	1.37c
30 min at RT	1.06	0.03	0.09	0.10	0.14	0.26	0.35	1.41m
60 min at RT	1.16	0.04	0.13	0.18	0.29	0.40	0.47	1.63a
N_2 Dry	1.18	0.06	0.09	0.10	0.11	0.15	0.19	1.37c

4 THE OPTIMIZED GRIT-BLAST/SILANE PROCESS

The goals of this investigation were to duplicate the silane surface preparation test results obtained by the Australian Aeronautical and Maritime Research Laboratory (DSTO) and determine the critical variables to optimize and control the process. The results clearly show that good wedge test performance at 120°F & 95-100% RH for 28 days, as exhibited by cohesive failure modes, can be obtained using the grit-blast/silane (GBS) surface preparation as long as key process variables are kept within reasonable operating windows.

4.1 Optimized GBS Process vs PAA

Phosphoric acid anodize (PAA) is perhaps the most durable surface preparation for aluminum. It is known that wedge test specimens prepared with PAA and primed with a corrosion-inhibiting primer will yield minimal crack growth and 100% cohesive failures when conditioned at 140°F & 95-100% RH for 28 days. Although this performance was not matched by GBS using FM 73 adhesive (see the additional data in Appendix A), the optimized GBS process compared favorably with PAA when the wedge test conditioning temperature was 120°F, as shown in Table 4.1-1. Failure modes for both panels were 100% cohesive. Deviation from the optimized process may still produce bonds which are "good enough" for a specific application but not as durable as those using the optimum process or PAA. However, there is no practical method for determining "good enough" for a prebond surface preparation.

Table 4.1-1 Wedge Test Comparison of PAA and GBS Surface Preparations

Surface Preparation	Initial (in)	Cumulative Crack Growth (in)						Total (in)
		1 hr	4 hr	8 hr	24 hr	7 days	28 days	
PAA	1.21	0.06	0.09	0.10	0.12	0.13	0.18	1.39c
GBS	1.06	0.05	--	0.08	0.09	0.10	0.13	1.19c

c: cohesive failure mode within the adhesive layer (95% or greater)

4.2 The Laboratory Grit-Blast/Silane (GBS) Process

The general outline for the GBS surface procedure, as optimized in the laboratory during this study, is shown below with discussion for each step.

• **Solvent Degrease**

This step removes gross contaminants, such as oils, from the surface to be treated. For this study, degreasing was accomplished by wiping the surface with clean wipes dampened with MEK. The GBS process should be fairly insensitive to the solvent since an aluminum oxide grit blast step follows all solvent applications. Alternate solvents were tested and found to be acceptable. The results were not reported because these evaluations were not conducted using controlled "soils" on the test panel adherends. The aluminum used in the study was very clean prior to preparation when compared to many situations encountered during repair of in-service components. The use of less effective solvents such as isopropyl alcohol (IPA) may not be suitable for many real-world repairs. If contamination is on the surface prior to grit-blasting, it could be embedded in the surface and adversely affect performance.

• **Scotch-Brite Abrade**

This step removes the oxide layer and solvent-resistant contaminants from the aluminum surface. In the UDRI effort, aluminum was hand abraded using 3M Company Scotch-Brite pads (7447) wet with MEK. Surfaces were abraded until "shiny" in appearance. It is also important to include an abrasion step prior to grit-blasting to ensure a consistent surface is presented each time the process is started so consistent results can be obtained. Grit-blasting directly without the abrasion step may be acceptable for some cases and not others, depending on the state of the adherends. Laboratory panels may not cause a problem, but surfaces soiled and otherwise affected by years in service may differ significantly from each other.

• **Solvent Clean**

This step removes the residual aluminum oxide and contaminants generated during the previous step. UDRI used clean, unpigmented/unscented wipes wet with MEK to clean the panels. Each wipe was used one time across the surface. Surfaces were solvent cleaned until no evidence of soil could be found on the wipes. A large number of wipes were required for this process.

• Aluminum Oxide Grit Blast

This step produces a macro-rough surface with greatly increased surface area. For this project, 50-micron aluminum oxide (low contaminant content) was used, except as noted. This is a very fine grit that is hard to contain when blasted. A portable blaster was used in a glove box. The pressure was supplied with dry, oil-free nitrogen, and the pressure hoses were dedicated for use with clean nitrogen. The aluminum received a light grit blast until it had a uniform, frosted appearance. There was a slight overlap with each pass, and at no time was the blaster allowed to dwell in one area. Spent grit was not reused. There was no attempt to quantify the nature of the blasted surface. All blasting was done to visual standards.

The pressure, standoff distance, and blaster angle to the surface was dependent on the operator. Nitrogen pressure to the spray gun was typically between 60 and 70 psi, and the standoff distance was approximately 6 inches from the aluminum surface. Since the circular nozzle of the blaster was relatively small (0.20in diameter), an angle to the surface of about 45° was used to increase the surface area exposed to the blast and prevent overblasting in a given area.

• Grit Removal

This step removes the residual aluminum oxide grit from the aluminum surface. In this study, dry, oil-free nitrogen at a pressure 70-80 psi was used to blast off the grit. No wiping or brushing was used to aid in the grit removal process. Some grit remained on the aluminum surfaces after the blasting was completed. No attempt was made to quantify the amount of residual grit that was left on the surface. Solvent wiping was not employed after grit-blasting since this can lead to contamination of the surface if improper rags, unclean solvent, or bad technique is used.

• Silane Hydrolysis

This step converts the silane coupling agent's siloxane groups into silanol groups which can couple with the oxide on the aluminum surface to be treated[2]. For the optimized process in this study, 1% by weight γ-GPS (Dow Corning Z-6040) in deionized water was continuously stirred in a glass beaker for 1 hour using a stir bar and magnetic mixer. The water pH was approximately 5.

• Silane Application

This step applies the hydrolyzed silane to the aluminum surface. In this study, unless otherwise noted, the aluminum panels were placed on a table in a horizontal position and "flooded" with the

silane solution. A camel's hair brush was used to keep the surface wet for 10 minutes; edges tended to dry. Brushing was accomplished from the center toward the edges in order to aid in the removal of residual aluminum oxide grit from the blasting operation. Additional silane was poured on the surface as required to keep it wet. During silane application, the surface was checked for water breaks. Any surfaces that were not water-break-free were reprocessed beginning with the first solvent degreasing step.

• <u>Silane Drying</u>

This step removes the excess silane solution from the aluminum surface and dries the surface at elevated temperature. It may also "cure" the applied coating by consolidating and crosslinking it. This was found to be critical for optimization of the GBS surface preparation during this study.

The majority of the data generated during this project (process variable studies in 1992-93) were obtained by drying in a preheated 200°F circulating air oven. Immediately after silane application, adherends were placed vertically on Duralace wipes, and excess solution was allowed to drain off. Then they were put (vertically) in racks and immediately place in the oven for one hour. In the 1998-99 silane drying study, the excess silane was blown off the panels with dry, oil-free nitrogen immediately following the 10-minute silane application. This was done using nitrogen pressure of approximately 30 psi, starting in the center and moving the nitrogen stream in a helical motion outward. This dried the panels with the least amount of streaking. The panels were then placed vertically in a rack and put into a preheated circulating air oven for one hour. Optimally, adherends were dried for one hour in the 210°F-230°F temperature range, according to the 1998-99 work. Both 200°F and 240°F drying temperatures (for one hour) produced results nearly as good (>90% cohesive failures). In earlier work, 100% cohesive failure modes were obtained when drying at 200°F. After drying, the aluminum adherends were cooled to ambient temperature by removal from the oven into the laboratory environment. Cure times other than one hour were not systematically investigated.

• <u>Priming</u>

This step, which follows the actual surface preparation, applies adhesive primer to the silane-treated aluminum. In this study, all treated aluminum was primed with BR 127 adhesive primer per Cytec's recommended practices via spraying with a conventional air gun using clean, dry nitrogen for pressure. Primer was precured prior to adhesive bonding. Dried primer thicknesses were in the 0.0001 to 0.0003 inch range (0.1-0.3 mil).

4.3 Repair Bonding Using the On-Aircraft Grit-Blast/Silane Process

Wedge test data comparable to those generated by UDRI have been obtained by at least four other organizations using on-aircraft procedures. The laboratory-optimized GBS process was slightly modified to meet the realities of on-aircraft application, primarily by WR-ALC/TI. Approximately 50 mechanics at WR-ALC/TI produced wedge test panels yielding cohesive failures with crack growth data similar to that obtainable using PAA (Appendix B). The mechanics fabricated wedge test panels using on-aircraft procedures in the repair environment as part of their certification process. The basics steps of the on-aircraft process are discussed below.

• **<u>Organic Coatings Removal</u>**

All organic coatings are removed from the area to be bonded prior to GBS surface preparation. This may be accomplished by any of several locally approved methods. Typically, chemical stripping or abrasion with 3M Company Scoth-Brite Roloc® discs is used. Plastic Media Blasting (PMB) should not be used since the plastic media can contaminate the bond surface.

• **<u>Solvent Degrease</u>**

After coatings are removed, a large area surrounding the repair location is degreased using MEK or acetone. Each succeeding step from degrease to silane application covers a smaller area, the last of which is slightly larger than the repair bonding location. This is done to minimize the chance of dragging contaminants into the repair area from the surrounding aircraft structure. Duralace aerospace wipes are used in the degreasing process because they are clean, lint-free, and unaffected by the solvent.

• **<u>Scotch-Brite Abrade</u>**

The abrasion step is completed using dry Scotch-Brite (or equivalent) pads as opposed abrading wet with solvent, as in the original laboratory procedure. Rather than hand abrading, the abrasive pads (such as 3-inch diameter very fine Scotch-Brite Roloc discs) are usually driven by a high-speed (20,000rpm) grinder. Pneumatic grinders are run using verified clean, dry nitrogen or air (about 90 psi) supplied through clean pressure hoses dedicated for this use. In addition to removal of the existing oxide layer and solvent-resistant contaminants, this step removes inorganic coatings, such as chemical conversion coatings. The aircraft aluminum is abraded until “shiny,” as in the laboratory process.

• Solvent Clean

The abraded area is solvent cleaned using MEK or acetone on Duralace wipes. Cleaning occurs until fresh wipes do not show signs of contaminant. Each wipe across the surface is not necessarily accomplished with a new wiper, as was the case for the laboratory work in 1992-93.

• Aluminum Oxide Grit Blast

Aluminum surfaces are grit-blasted in the same manner as in the laboratory process, using verified clean, dry nitrogen or air with dedicated pressure hoses. Extreme care is taken to contain and clean up the aluminum oxide grit since it can be detrimental to aircraft systems. The ability of the fine grit to remain airborne and find its way throughout the work area cannot be overestimated, nor can the difficulty and care required to adequately contain it and clean-up.

• Grit Removal

Removal of excess grit is conducted in a manner similar to the laboratory procedure. Solvent wiping is not used. The surface is blown off with verified clean, dry nitrogen or air at about 70-80 psi. This may be assisted by wiping (dry) with a soft-bristle brush.

• Silane Hydrolysis

Timing of the hydrolysis step is more difficult for on-aircraft repair scenarios than it is in a laboratory. The silane must be ready immediately after grit-blasting to allow the repair to proceed, but it must not be ready so early that the maximum acceptable hydrolysis time is exceeded. In the laboratory, application occurred within minutes after completion of the 1-hour hydrolysis time. During aircraft surface preparation, the silane is not allowed to hydrolyze more than 4 hours, with 1 hour as the goal. Mixing the silane/water solution by volume, using premeasured 1-ml quantities of silane stored in sealed vials, makes the process easier. A graduated cylinder is used to measure the water into a glass or polypropylene beaker. For each 150ml of water, two of the 1-ml vials of silane are used. This gives a silane solution that is safely in the optimal 1-3% range. The water source, both from contamination and pH perspectives, is important. Distilled or deionized water is used. If necessary, the pH can be adjusted (to the 4-6 range) using acetic acid; a portable pH meter is useful for this operation.

• Silane Application

Silane application on aircraft is accomplished using brushes, as in the laboratory procedure. Clean acid brushes are typically used. Since the surface is not likely to be horizontal, flooding by pouring

on silane is not practical. On both horizontal and vertical surfaces, continual brushing for 10 minutes keeps the surface wet with silane. As in the lab, brushing is from the center outward to help remove remaining aluminum oxide grit. Some grit remains on the surface even after silane brush application. The amount of "acceptable" residual grit has not been quantified.

• **Excess Silane Solution Removal**

After on-aircraft silane application, there is typically a significant delay before heat can be brought to the surface. In order to prevent the silane from slowly drying on the surface at ambient conditions, it is blown off using verified clean, dry nitrogen or air. This again is done from the center outward. If done properly, streaking is minimized.

• **Silane Drying**

Heating of the silane on aircraft can be done in a number of ways as long as the desired temperature (nominally 220°F) can be maintained for one hour, within acceptable tolerances and without contaminating the surface. This can be quite difficult for some applications since temperature ranges over 30°F can be encountered due to structural "heat sinks." In all cases, the heating apparatus should not touch the surface. Multiple-bulb infrared heat lamps are often used for this step. Temperature control is maximized when power settings for individual bulbs can be separately controlled. Thermal surveys of the structure are helpful prior to start of the surface preparation to ensure the heating technique will enable the repair area to reach the requisite temperature as well as to be certain the temperature can be accurately monitored. Usually thermocouples surrounding the repair area are used to monitor temperature; their relationship to the actual repair area must be understood. Insulation is often necessary around the repair area or on the backside, if accessible. When using heat lamps, care is taken to make sure thermocouples are not being directly heated by the lamps so the temperature readings are truly those of the aluminum surface. Time to set up the heating apparatus and monitoring thermocouples is significant. These tasks are accomplished as quickly as possible since the delay between silane application and the start of heating should be minimized and in no case should exceed one hour.

• **Priming**

After the aircraft surface is cooled to 90°F or less, the repair area is primed with Cytec BR 127 CIAP which is cured prior to adhesive application. Primer may be applied by spray or brush technique. Cure is typically conducted using infrared heat lamps to bring the primed metal surface to a minimum of 200°F for 90 minutes. Clean, dry nitrogen or air is used for spraying.

5 CONCLUSIONS

The conditions necessary to establish a grit-blast/silane (GBS) surface preparation as a viable option for aluminum treatment prior to structural adhesive bonding have been established. The various processing parameters for the silane surface preparation were evaluated using the wedge test (ASTM D 3762), since it is considered a key indicator of bonded joint long-term environmental durability and has been used to optimize other surface preparation processes.

Attempts made to duplicate wedge test results reported by the Australian Defence Science and Technology Organisation (DSTO) for their silane surface preparation were initially unsuccessful. This inability to duplicate the DSTO results had been experienced by several other organizations and was apparently due to an incomplete understanding of the DSTO process. After failure to obtain the desired results, and without detailed knowledge of the DSTO process, an effort was successfully undertaken to optimize the GBS surface preparation, based on the general DSTO approach. The optimized process, believed to be similar to DSTO's procedure, yields wedge test data comparable to those obtained from bonded joints using the state-of-the-art aluminum prebond surface preparation, phosphoric acid anodize (PAA). Wedge test failure modes for the optimized process are generally cohesive within the adhesive layer (not involving the silane surface preparation) when conditioned at 120°F & 95-100% RH for 28 days.

Several parameters were identified and studied during the program. These included: silane drying time and temperature; silane solution concentration; water pH; hydrolysis time; application time; and aluminum oxide grit size. Other variables that affect practical application of the GBS process were also evaluated. Optimal values and acceptable tolerances were established for each parameter. Elevated-temperature drying conditions for the silane on the treated aluminum surface, optimal between 210°F and 230°F for one hour, appeared to be the single most important variable. The detailed procedure for the AFRL/MLSA optimized grit-blast/silane (GBS) surface preparation for on-aircraft bonded repair application is given in Appendix D.

Despite the considerable testing and successful results obtained in this study, additional work would be helpful in order to fully understand and improve silane surface preparation processes. This includes: 1) assessing performance by means other than the wedge test; 2) studying interactions between various process parameters in a systematic way; 3) characterizing the morphology and chemistry of silane-treated surfaces; 4) determining the mechanism(s) which enables the surface preparation to produce good adhesion and durability performance; and 5) evaluating silanes other than the epoxy-functional γ-GPS as well as hydrolysis in media other

than pure water. Various organizations have already contributed significantly in one or more of these areas[27,28,29]. With their work, a more robust silane surface preparation process is possible.

AFRL/MLSA still considers PAA the premier aluminum prebond surface preparation; much data have been generated using this process, and it has a long history of success in a variety of actual bonded joint applications. PAA/primer should still be the first choice for aluminum prebond surface preparation when the optimal (dip-tank) process can be applied. Although nontank versions of PAA exist, it is not convenient or even possible to use these for many on-aircraft repair applications. Phosphoric acid has potential to embrittle certain high-strength steel fasteners that may be in the repair area[30], and residual acid could lead to corrosion of aluminum structure if not completely removed. In many organizations, acids pose a hazardous materials problem requiring special tracking, handling, and disposal procedures. For some applications, the PAA electrical power requirement presents a safety concern. Many applications are outside the optimal process temperature range. Finally, repair area geometry and accessibility can limit the use of on-aircraft PAA processes.

GBS provides a high-performance, nonacid option for on-aircraft surface preparation of aluminum for adhesive bonding. The process has also shown potential for use on certain steel and titanium alloys. Operating windows for the key processing parameters defined in this study have proven to be wide enough to control the process and enable successful repair applications. GBS was initially implemented in the U.S. Air Force at WR-ALC to solve a critical need for the C-141 aircraft in 1993[31]. Good wedge test performance was a major consideration in the selection of GBS for the C-141 application, even though testing was conducted at 120°F as opposed to the desired 140°F. Risk was mitigated by the fact the RAAF claimed excellent service performance with their similar silane surface preparation. It appears that GBS produces acceptable bond strength and durability performance, as evidenced by its nearly 10-year service history in the U.S. Air Force for thousands of bonded repairs[32]. As additional service experience is obtained, the 120°F wedge test requirement could become the standard for assessing adequate surface preparation performance.

Despite its advantages, GBS is not the ideal on-aircraft process, primarily due to the requirements for grit-blasting and elevated-temperature silane drying. These steps take considerable time and present safety concerns for personnel and the aircraft. Recent work sponsored by AFRL/MLSA on a similar surface preparation using more reactive sol-gel chemistry has the potential to eliminate some of the GBS disadvantages[33,34,35]. As is always the case, the best prebond surface preparation approach must be selected for each application after considering the merits of the available candidate procedures.

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APPENDIX A:

ADDITIONAL GBS DATA

Introduction

This appendix contains miscellaneous GBS data (Table A-1 through Table A-10) that were generated after the effort that constitutes the bulk of this report. These data do not necessarily directly relate to anything discussed in the body of the report. They include: data for 3M Company AF 163-2 adhesive; GBS wedge tests at 140°F; data for bonding under partial vacuum as opposed to positive pressure; and data for GBS utilizing Cytec BR 6747-1 waterborne primer rather than BR 127. The results of scuff-sand/silane testing are also included to demonstrate the need for the grit-blast step of the GBS procedure.

Along with the additional wedge test data of Tables A-1 through A-6), GBS lap shear and peel data are provided along with PAA/BR 127 data for comparison purposes. These data have been added to demonstrate the GBS process provides lap shear and peel strengths comparable to those obtained using the PAA surface preparation technique. Failure modes were defined differently for these specimens than they were for wedge tests, as indicated on the applicable tables (Table A-7 through Table A-10).

Table A-1 GBS Data with FM 73 Adhesive for Various Silane Drying Temperatures, Cured Under Positive Pressure, Tested at 140°F & 95-100% RH

Drying Time and Temperature	Initial (in)	Cumulative Crack Growth (in)					Total (in)
		1 hr	8 hr	24 hr	7 days	28 days	
1 hr at 200°F	1.13	0.02	0.10	0.15	0.20	0.30	1.43a
1 hr at 220°F	1.16	0.05	0.11	0.16	0.23	0.32	1.48a
1 hr at 230°F	1.13	0.03	0.11	0.15	0.21	0.34	1.47a

a: adhesive failure mode (interfacial at the metal surface)

Table A-2 GBS Data with AF 163-2 Adhesive for Various Silane Drying Temperatures, Cured Under Positive Pressure, Tested at 140°F & 95-100% RH

Drying Time and Temperature	Initial (in)	Cumulative Crack Growth (in)					Total (in)
		1 hr	8 hr	24 hr	7 days	28 days	
1 hr at 175°F	1.25	0.06	0.14	0.21	0.32	0.41	1.66a
1 hr at 200°F	1.20	0.04	0.11	0.15	0.26	0.36	1.56a
1 hr at 220°F	1.18	0.04	0.11	0.15	0.25	0.32	1.50a
1 hr at 230°F	1.17	0.04	0.12	0.16	0.23	0.32	1.49a

a: adhesive failure mode (interfacial at the metal surface)

Table A-3 GBS Data with AF 163-2 Adhesive for a Silane Drying Temperature of 200°F, Cured Under Positive Pressure, Tested at 120°F & 95-100% RH

Drying Time and Temperature	Initial (in)	Cumulative Crack Growth (in)					Total (in)
		1 hr	8 hr	24 hr	7 days	28 days	
1 hr at 200°F	1.21	0.07	0.07	0.10	0.13	0.15	1.36c
1 hr at 200°F	1.26	0.01	0.04	0.05	0.07	0.11	1.37c
1 hr at 200°F	1.20	0.08	0.10	0.11	0.12	0.16	1.36c
1 hr at 200°F	1.10	0.06	0.08	0.08	0.11	0.12	1.22c
1 hr at 200°F	1.09	0.11	0.14	0.15	0.18	0.20	1.29c

c: cohesive failure mode within the adhesive layer (95% or greater)

Table A-4 GBS Data with AF 163-2 Adhesive for a Silane Drying Temperature of 200°F, Cured Under Partial Vacuum, Tested at 120°F & 95-100% RH

Drying Time and Temperature	Initial (in)	Cumulative Crack Growth (in)					Total (in)
		1 hr	8 hr	24 hr	7 days	28 days	
1 hr at 200°F	1.29	0.04	0.07	0.08	0.12	0.13	1.42c
1 hr at 200°F	1.24	0.04	0.04	0.06	0.10	0.11	1.35c

c: cohesive failure mode (> 95% within the adhesive layer)

Table A-5 GBS Data with AF 163-2 Adhesive Primed using BR 6747-1 Primer

Test Temp.	Initial (in)	Cumulative Crack Growth (in)					Total (in)
		1 hr	8 hr	24 hr	7 days	28 days	
120°F	1.16	0.03	0.06	0.09	0.13	0.16	1.32c
140°F	1.14	0.04	0.12	0.17	0.22	0.26	1.40a

a: adhesive failure mode (interfacial at the metal surface)

c: cohesive failure mode within the adhesive layer (95% or greater)

Table A-6 Scuff-Sand/Silane Data with AF 163-2 Adhesive for both Partial Vacuum (PV) and Positive Pressure (PP) Cure Pressures and Primer vs. No Primer; Tested at 120°F & 95-100% RH

Cure Pressure	Primer	Initial (in)	Cumulative Crack Growth (in)					Total (in)
			1 hr	8 hr	24 hr	7 days	28 days	
PV	None	1.23	0.08	0.15	0.28	0.47	0.55	1.78a
PV	None	1.28	0.16	0.24	0.33	0.56	0.59	1.87a
PV	BR 127	1.31	0.27	0.38	0.48	0.65	0.72	2.03a
PV	BR 127	1.28	0.41	0.55	0.73	1.00	1.08	2.36a
PP	None	1.26	0.09	0.15	0.30	0.49	0.50	1.76a
PP	None	1.25	0.13	0.22	0.31	0.53	0.64	1.89a
PP	BR 127	1.28	0.47	0.63	0.82	0.95	0.97	2.25a
PP	BR 127	1.26	0.41	0.47	0.58	0.73	0.77	2.03a

a: adhesive failure mode (interfacial at the metal surface)

Table A-7 GBS Tensile Lap Shear Data for 2024-T3 Aluminum Panels Primed with BR 127 and Bonded with 3M AF 163-2; tested at 70°F

Cure Pressure	Cure Cycle	GLT (in)	Average Strength (psi)	Failure Mode
Partial Vacuum	1 hr @ 250°F @ 20 in Hg	0.005	1825	Mixed
Partial Vacuum	1 hr @ 250°F @ 20 in Hg	0.004	3054.8	Mixed
Partial Vacuum	1 hr @ 250°F @ 20 in Hg	0.004	2923.1	Mixed
Positive Pressure	1 hr @ 250°F @ 35 psi	0.005	5555	Mixed
Positive Pressure	1 hr @ 250°F @ 35 psi	0.003	5306	Mixed

Mixed failure mode: greater than 10% cohesive but less than 95% cohesive.

Table A-8 PAA Tensile Lap Shear Data for 2024-T3 Aluminum Panels Primed with BR 127 and Bonded with 3M AF 163-2; tested at 70°F

Cure Pressure	Cure Cycle	GLT (in)	Average Strength (psi)	Failure Mode
Partial Vacuum	1 hr @ 250°F @ 12 in Hg	0.004	5326.0	Mixed
Partial Vacuum	1 hr @ 250°F @ 20 in Hg	0.003	3689.0	Mixed
Partial Vacuum	1 hr @ 250°F @ 20 in Hg	0.002	4258.0	Mixed
Positive Pressure	1 hr @ 250°F @ 35 psi	0.004	5937.0	Cohesive
Positive Pressure	1 hr @ 250°F @ 35 psi	0.004	5680.0	Cohesive

Mixed failure mode: greater than 10% cohesive but less than 95% cohesive.
Cohesive failure mode: greater than 95% cohesive.

Table A-9 GBS Floating Roller Peel Data for 2024-T3 Aluminum Panels Primed with BR 127 and Bonded with 3M AF 163-2; tested at 70°F

Cure Pressure	Cure Cycle	GLT (in)	Average Strength (pli)	Failure Mode
Partial Vacuum	1 hr @ 250°F @ 20 in Hg	0.007	62.0	Cohesive
Positive Pressure	1 hr @ 250°F @ 35 psi	0.006	65.0	Mixed

Mixed failure mode: greater than 10% cohesive but less than 95% cohesive.
Cohesive failure mode: greater than 95% cohesive.

Table A-10 PAA Floating Roller Peel Data for 2024-T3 Aluminum Panels Primed with BR 127 and Bonded with 3M AF 163-2; tested at 70°F

Cure Pressure	Cure Cycle	GLT (in)	Average Strength (pli)	Failure Mode
Partial Vacuum	1 hr @ 250°F @ 20 in Hg	0.007	61.0	Mixed
Partial Vacuum	1 hr @ 250°F @ 20 in Hg	0.006	61.0	Mixed
Positive Pressure	1 hr @ 250°F @ 35 psi	0.009	60.0	Mixed
Positive Pressure	1 hr @ 250°F @ 35 psi	0.007	47.0	Mixed

Mixed failure mode: greater than 10% cohesive but less than 95% cohesive.

APPENDIX B

WR-ALC MECHANICS' WEDGE TEST DATA

Introduction

The data in this appendix were generated by Warner Robins Air Logistics Center (WR-ALC) mechanics as part of their certification required prior to conducting on-aircraft repairs using the GBS process. The wedge test panels were fabricated under repair conditions in the aircraft maintenance hangar "under the shadow of the aircraft." Adherends were all 7075-T6 bare aluminum. The GBS process was conducted using on-aircraft equipment, including infrared heat lamps for silane drying and BR 127 primer cure. The surface was dry abraded using fine Scotch-Brite Roloc discs on die grinders (driven by nitrogen). The excess silane solution was blown off of the aluminum adherends using clean, dry nitrogen immediately after the 10-minute application time and just prior to 200±5°F drying for one hour. Adhesives were all 0.085 psf with knit carrier. Adhesive cure was under a vacuum bag (approximately 21 in Hg) for 60 minutes at 250±5°F after a heat-up rate of 3-5°F per minute using hot bonder controllers and electrical resistance heat blankets. AFRL/MLSA machined the wedge test panels into specimens and tested them at 120°F & 95-100% RH for 7 days. The certification criteria was cohesive failure (100% for all specimens) after 24 hours of conditioning. Maximum allowable crack extension was 0.20 inch. This value was selected solely because it ensured cohesive failures at 24 hours of conditioning, so failure mode was really the only pass/fail criterion. The 0.085 psf (versus 0.06 psf) adhesive and vacuum bag cures (versus positive pressure) led to slightly thicker bondlines with significantly more porosity than seen during the silane study conducted by AFRL/MLSA. For these reasons, the WR-ALC wedge tests may have been a bit less stringent than those tested during the AFRL/MLSA study. The data are presented in Table B-1, Table B-2, and Table B-3.

Table B-1 WR-ALC Certification Wedge Test Panels – November 1993

Mechanic	Adhesive	Initial (in)	Cumulative Crack Growth (in) 1 hr	4 hr	8 hr	24 hr	7 days	Total (in)
RGT	FM 73	1.11	0.07	0.09	0.12	0.18	0.21	1.32c
MM	FM 73	1.11	0.05	0.08	0.09	0.16	0.17	1.28c
TH	FM 73	1.10	0.07	0.10	0.12	0.16	0.17	1.27c
VG	FM 73	1.11	0.09	0.10	0.12	0.19	0.43	1.54a
HC	FM 73	1.14	0.04	0.06	0.08	0.14	0.18	1.32c
S	FM 73	1.08	0.07	0.08	0.09	0.15	0.19	1.27c
DT	FM 73	1.14	0.04	0.06	0.06	0.11	0.25	1.39a
MV	FM 73	1.14	0.02	0.04	0.06	0.08	0.16	1.30c
CC	FM 73	1.08	0.06	0.08	0.09	0.11	0.16	1.24c
RH	FM 73	1.09	0.15	0.23	0.25	0.35	0.41	1.50a
GW	FM 73	1.12	0.07	0.12	0.14	0.15	0.20	1.32c
AS	FM 73	1.07	0.07	0.09	0.09	0.12	0.18	1.25c
VD	FM 73	1.16	0.05	0.08	0.09	0.10	0.16	1.32c
Cy	FM 73	1.09	0.05	0.05	0.05	0.06	0.13	1.22c
TH	AF 163-2K	1.12	0.05	0.07	0.08	0.11	0.16	1.28c
TB	AF 163-2K	1.10	0.05	0.07	0.11	0.15	0.17	1.27c
BC	AF 163-2K	1.14	0.06	0.07	0.09	0.11	0.13	1.27c
HB	AF 163-2K	1.10	0.02	0.06	0.09	0.11	0.15	1.25c
B	AF 163-2K	1.09	0.04	0.07	0.10	0.14	0.18	1.27c
J	AF 163-2K	1.09	0.03	0.03	0.06	0.10	0.16	1.25c
S	AF 163-2K	1.18	0.05	0.08	0.09	0.11	0.19	1.37c
LJ	EA 9628	1.37	0.03	0.05	0.05	0.06	0.07	1.44c

a: adhesive failure mode at the metal interface with the primer
c: cohesive failure mode within the adhesive layer (95% or greater)

Table B-2 WR-ALC Certification Wedge Test Panels – December 1993

Mechanic	Adhesive	Initial (in)	Cumulative Crack Growth (in) 1 hr	4 hr	8 hr	24 hr	7 days	Total (in)
DT2	FM 73	1.14	0.04	0.06	0.06	0.11	0.14	1.28c
V	FM 73	1.12	0.08	0.09	0.10	0.11	0.13	1.25c
JG	FM 73	1.13	0.05	0.07	0.07	0.11	0.12	1.25c
MMc	FM 73	1.11	0.05	0.07	0.07	0.10	0.11	1.22c
DC	FM 73	1.17	0.08	0.12	0.12	0.14	0.15	1.32c
MC	FM 73	1.06	0.07	0.09	0.12	0.13	0.13	1.19m
JS	FM 73	1.14	0.07	0.09	0.11	0.12	0.13	1.27c
WW	FM 73	1.07	0.09	0.10	0.12	0.14	0.16	1.23c
MW	FM 73	1.15	0.07	0.08	0.08	0.11	0.12	1.27c
TJ	FM 73	1.16	0.06	0.07	0.08	0.10	0.12	1.28c
RE	FM 73	1.14	0.06	0.07	0.08	0.09	0.13	1.27c
WD	FM 73	1.13	0.10	0.11	0.14	0.14	0.17	1.30c
SC	FM 73	1.17	0.13	0.15	0.22	0.25	0.33	1.50a
SB	FM 73	1.19	0.07	0.08	0.09	0.11	0.16	1.35c
DS	FM 73	1.13	0.06	0.09	0.09	0.11	0.15	1.28c
JP	FM 73	1.09	0.09	0.09	0.11	0.12	0.17	1.26c
DK	FM 73	1.12	0.08	0.09	0.12	0.12	0.16	1.28c
FH	FM 73	1.15	0.05	0.05	0.07	0.07	0.16	1.31c
LAJ	FM 73	1.13	0.04	0.05	0.09	0.10	0.14	1.27c
HM	FM 73	1.10	0.06	0.07	0.09	0.11	0.15	1.25c
H	FM 73	1.10	0.07	0.08	0.09	0.11	0.15	1.25c
M	FM 73	1.12	0.04	0.06	0.07	0.08	0.13	1.25c
RH2	FM 73	1.12	0.04	0.06	0.08	0.12	0.16	1.28m
GF	FM 73	1.12	0.09	0.12	0.18	0.50	1.00	2.12a
Co	FM 73	1.12	0.04	0.05	0.07	0.10	0.12	1.24c
SC2	FM 73	1.17	0.07	0.07	0.10	0.14	0.17	1.34c

a: adhesive failure mode at the metal interface with the primer
c: cohesive failure mode within the adhesive layer (95% or greater)
m: mixed failure mode with some specimens exhibiting interfacial failure and others having cohesive failure within the adhesive layer

Table B-3 WR-ALC Certification Wedge Test Panels – May 1994

Mechanic	Adhesive	Initial (in)	Cumulative Crack Growth (in)					Total (in)
			1 hr	4 hr	8 hr	24 hr	7 days	
Ca	FM 73	1.10	0.06	0.10	0.12	0.14	0.16	1.26c
TR	FM 73	1.13	0.06	0.08	0.11	0.15	0.15	1.28c
RJ	FM 73	1.13	0.07	0.10	0.11	0.16	0.21	1.34m
CB	FM 73	1.16	0.08	0.11	0.13	0.16	0.42	1.58a
TH	FM 73	1.13	0.05	0.10	0.13	0.15	0.16	1.29c
JN	FM 73	1.09	0.04	0.07	0.09	0.09	0.10	1.29c
HK	FM 73	1.16	0.08	0.11	0.14	0.17	0.35	1.51a

a: adhesive failure mode at the metal interface with the primer
c: cohesive failure mode within the adhesive layer (95% or greater)
m: mixed failure mode with some specimens exhibiting interfacial failure and others having cohesive failure within the adhesive layer

APPENDIX C

ADDITIONAL SILANE DRYING TEMPERATURE DATA

Introduction

Silane drying at various temperatures was evaluated on several occasions using the wedge test. In the original UDRI study conducted in 1992-93, the optimal drying temperature was determined to be 200°F, and durability results were degraded for temperatures over 210°F. In 1998, it was discovered that the RAAF was drying silane at temperatures well above the maximum acceptable temperature indicated by the UDRI study. Their purpose was to reduce porosity in bondlines caused by moisture on adherend surfaces during vacuum bag cures. Published data from DSTO indicated the need to dry the treated adherends at 110°C (230°F) to minimize porosity. Additional silane drying work was conducted by UDRI and AFRL/MLSA in 1998-99 to verify their original study since it contradicted the DSTO work and RAAF practices. The results of the new study were more in line with the DSTO results and revealed an optimum drying temperature of 220°F, with fairly good wedge test results obtained when drying up to 240°F. The 200°F drying results in the second study were not as good as the "optimum" results (100% cohesive failures) determined for that temperature in 1992-93. The failure modes in the 1998-99 study almost met the criterion for "cohesive" but were considered "adhesive." The specimens exhibited less than 95% (but typically more than 90%) cohesive failure, with a small amount of interfacial failure away from the edges.

Additional drying studies were conducted to resolve the discrepancies. These evaluations led to disappointing and inconsistent results for all temperatures tested. In fact, for undetermined reasons, 100% cohesive failure modes could not be achieved for the GBS process on wedge test panels produced by UDRI, AFRL/MLSA, and WR-ALC/EN laboratory personnel. Between each trial, factors that may have been causing the problem were considered. These included water pH as well as silane and BR 127 primer age. No solution was found. Better understanding of the GBS process is apparently required to keep it in control. Perhaps it is not as robust as first thought. However, AFRL/MLSA and UDRI fabricated hundreds of wedge test specimens with cohesive failure modes (typically 100%) between 1993 and 1998 using the 200°F drying step. Other organizations have also successfully used the process over the past 5-7 years. WR-ALC mechanics continue to produce passing wedge test panels with 200°F silane drying as part of their periodic and ongoing recertification process.

The data below are included in this report for completeness. If they had not been repeated, they would have been excluded as anomalous. The GBS surface preparation, even with silane drying at 200°F, is still considered an excellent aluminum on-aircraft prebond surface preparation as evidenced by its flawless in-service record. It is still employed by WR-ALC on a continual basis for on-component repairs both on the flight line and in the bond shop.

Table C-1 represents specimens made by UDRI and AFRL/MLSA personnel. Specimens labeled “A” and “B” were made by the same individual. Those labeled “C” and “D” were made by another. The third person fabricated specimens for only the 200°F and 220°F cases. These are labeled “E” and “F” in the table. Table C-2 and Table C-3 represent the next attempts to evaluate silane drying temperature. No panels yielded cohesive failures for all specimens. The results for the final silane drying evaluations are presented in Table C-4. The panels were treated by WR-ALC/EN personnel, with bonding and testing conducted by UDRI. Nearly all test panels resulted in adhesive (interfacial) or mixed mode failures.

The data reported in all four tables were generated from specimens prepared in a manner similar to that used during the original 1992-93 UDRI effort. One difference was the fact the Scotch-Brite abrade procedure was not conducted wet with MEK. UDRI’s later work used hand pads (Scotch-Brite 7447) and abraded with Alconox® detergent and water. This was followed by water rinsing, drying, and an acetone wipe. The WR-ALC abrasion process (2003 study) utilized Scotch-Brite Roloc discs rather than hand pads and was conducted dry followed by solvent wiping with MEK. The aluminum alloy was 2024-T3 bare and the adhesive was Cytec FM 73 (0.06 psf with a knit carrier) for all cases. Water pH was about 5 for these tests.

All data in the tables represent one wedge test panel (the average crack lengths of the 5 specimens in the panel). Much of the data show “mixed” failure modes. This means at least one specimen, and sometimes four of five specimens, in a panel exhibited a cohesive failure mode of 95% or greater. These data clearly contradict the oft-stated adage that most crack extension in the wedge test occurs within the first hour*. This may have been true of process control wedge test specimens for sulfuric acid-sodium dichromate and PAA, but is clearly not the case for the GBS data in this report. The 200°F drying data in Table C-4 is a clear example of this.

* “Standard Test Method for Adhesive-Bonded Surface Durability of Aluminum (Wedge Test),” ASTM D 3762-79, Volume 15.06 - Adhesives, American Society for Testing and Materials, Philadelphia PA, 1997, p 256.

Table C-1 Silane Drying Temperature Evaluation – February 2001

Dry Temperature		Initial (in)	Cumulative Crack Growth (in) 1 hr	8 hr	24 hr	7 days	28 days	Total (in)
150°F	A	1.03	0.04	0.115	0.15	0.20	0.26	1.29a
	B	1.13	0.02	0.14	0.17	0.27	0.42	1.55a
	C	1.15	0.04	0.10	0.10	0.19	0.37	1.52a
	D	1.16	0.05	0.10	0.11	0.20	0.38	1.54a
160°F	A	1.08	0.02	0.10	0.11	0.18	0.20	1.28a
	B	0.98	0.01	0.14	0.14	0.14	0.18	1.16m
	C	1.09	0.05	0.10	0.11	0.17	0.22	1.31a
	D	1.14	0.06	0.09	0.09	0.14	0.16	1.30m
170°F	A	1.04	0.03	0.13	0.15	0.19	0.23	1.27m
	B	1.02	0.02	0.13	0.14	0.19	0.20	1.22m
	C	1.08	0.07	0.09	0.12	0.14	0.15	1.23c
	D	1.08	0.06	0.09	0.12	0.14	0.18	1.26c
180°F	A	1.02	0.01	0.13	0.14	0.19	0.23	1.25m
	B	1.05	0.03	0.12	0.14	0.19	0.23	1.28a
	C	1.09	0.07	0.10	0.13	0.16	0.18	1.27c
	D	1.01	0.07	0.10	0.12	0.16	0.16	1.17c
190°F	A	1.17	0.08	0.11	0.12	0.15	0.20	1.37m
	B	1.17	0.07	0.10	0.12	0.14	0.21	1.38m
	C	1.10	0.06	0.10	0.12	0.14	0.18	1.28c
	D	1.06	0.06	0.10	0.11	0.13	0.16	1.22c
200°F	A	1.10	0.02	0.03	0.05	0.08	0.09	1.19c
	B	1.13	0.03	0.06	0.07	0.07	0.11	1.24c
	C	1.07	0.06	0.07	0.08	0.10	0.13	1.20c
	D	1.08	0.03	0.06	0.07	0.12	0.14	1.22c
	E	1.12	0.08	0.13	0.13	0.31	0.39	1.51a
	F	1.04	0.07	0.12	0.13	0.16	0.19	1.23m
210°F	A	1.07	0.04	0.10	0.10	0.15	0.21	1.28a
	B	1.13	0.04	0.10	0.12	0.23	0.44	1.57a
	C	1.06	0.04	0.08	0.09	0.15	0.20	1.26a
	D	1.09	0.04	0.10	0.12	0.16	0.21	1.30a
220°F	A	1.25	0.05	0.07	0.07	0.08	0.10	1.35m
	B	1.22	0.04	0.07	0.10	0.10	0.11	1.33m
	C	1.08	0.04	0.11	0.13	0.19	0.21	1.29a
	D	1.08	0.04	0.11	0.13	0.17	0.20	1.28m
	E	1.05	0.06	0.12	0.13	0.18	0.21	1.26m
	F	1.14	0.05	0.09	0.12	0.17	0.28	1.42m
230°F	A	1.16	0.03	0.07	0.09	0.14	0.29	1.45a
	B	1.18	0.03	0.09	0.10	0.18	0.38	1.56a
	C	1.11	0.03	0.05	0.08	0.11	0.13	1.24m
	D	1.08	0.04	0.08	0.09	0.15	0.18	1.26m
240°F	A	1.11	0.05	0.08	0.11	0.14	0.18	1.29a
	B	1.14	0.04	0.06	0.09	0.12	0.17	1.31a
	C	1.10	0.04	0.09	0.10	0.15	0.21	1.31a
	D	1.08	0.02	0.05	0.08	0.13	0.14	1.22a

a: adhesive failure mode at the metal interface with the primer
c: cohesive failure mode within the adhesive layer
m: mixed failure mode with some specimens exhibiting interfacial failure and others having cohesive failure within the adhesive layer

Table C-2 Silane Drying Temperature Evaluation – September 2001

Silane Drying Temperature	Initial (in)	Cumulative Crack Growth (in)					Total (in)
		1 hr	8 hr	24 hr	7 days	28 days	
150°F	1.17	0.02	0.06	0.07	0.13	0.40	1.57a
160°F	1.12	0.03	0.07	0.08	0.15	0.37	1.49a
170°F	1.12	0.03	0.08	0.09	0.11	0.26	1.38m
180°F	1.15	0.02	0.08	0.08	0.12	0.31	1.46m
190°F	1.11	0.07	0.11	0.11	0.18	0.40	1.51a
200°F	1.14	0.04	0.05	0.07	0.17	0.26	1.40a
210°F	1.10	0.04	0.08	0.08	0.15	0.22	1.32m
220°F	1.15	0.04	0.08	0.10	0.16	0.21	1.36m
230°F	1.16	0.04	0.07	0.08	0.13	0.21	1.37m
240°F	1.11	0.04	0.07	0.09	0.14	0.23	1.34m

a: adhesive failure mode at the metal interface with the primer
m: mixed failure mode with some specimens exhibiting interfacial failure and others having cohesive failure (>95%) within the adhesive layer

Table C-3 Silane Drying Temperature Evaluation – October 2001

Silane Drying Temperature	Initial (in)	Cumulative Crack Growth (in)					Total (in)
		1 hr	8 hr	24 hr	7 days	28 days	
150°F	1.09	0.03	0.06	0.07	0.13	0.15	1.24m
160°F	1.10	0.02	0.07	0.08	0.15	0.31	1.41a
170°F	1.17	0.05	0.07	0.09	0.27	0.44	1.61a
180°F	1.13	0.04	0.08	0.09	0.16	0.17	1.30m
190°F	1.06	0.02	0.06	0.06	0.13	0.18	1.24m
200°F	1.15	0.01	0.06	0.08	0.18	0.27	1.42a
210°F	1.15	0.03	0.06	0.06	0.14	0.15	1.30m
220°F	1.10	0.01	0.04	0.08	0.15	0.21	1.31m
230°F	1.15	0.02	0.05	0.09	0.25	0.37	1.52m
240°F	1.11	0.04	0.06	0.07	0.17	0.20	1.31m

a: adhesive failure mode at the metal interface with the primer
m: mixed failure mode with some specimens exhibiting interfacial failure and others having cohesive failure (>95%) within the adhesive layer

Table C-4 Final Silane Drying Temperature Evaluation – March 2003

Dry Temperature		Initial (in)	Cumulative Crack Growth (in)					Total (in)
			1 hr	8 hr	24 hr	7 days	28 days	
160°F	1	1.12	0.02	0.05	0.12	0.22	0.45	1.57a
	2	1.14	0.01	0.02	0.10	0.24	0.51	1.65a
	3	1.06	0.03	0.08	0.11	0.19	0.40	1.46a
170°F	1	1.08	0.03	0.03	0.08	0.46	0.22	1.30m
	2	1.06	0.00	0.05	0.10	0.20	0.41	1.47a
	3	1.09	0.03	0.07	0.09	0.14	0.22	1.31m
180°F	1	1.09	0.00	0.05	0.11	0.18	0.23	1.32m
	2	1.10	0.01	0.04	0.13	0.18	0.27	1.37m
	3	1.07	0.04	0.06	0.10	0.19	0.21	1.28m
190°F	1	1.09	0.04	0.06	0.12	0.26	0.43	1.52a
	2	1.08	0.03	0.07	0.11	0.19	0.26	1.34m
	3	1.06	0.04	0.09	0.12	0.22	0.44	1.50a
200°F	1	1.09	0.00	0.03	0.10	0.49	0.70	1.79a
	2	1.07	0.02	0.04	0.08	0.20	0.35	1.42m
	3	1.13	0.04	0.05	0.06	0.17	0.54	1.67a
210°F	1	1.08	0.02	0.04	0.09	0.15	0.23	1.31m
	2	1.08	0.03	0.06	0.10	0.15	0.26	1.34m
	3	1.11	0.04	0.05	0.07	0.21	0.40	1.51a
220°F	1	1.07	0.03	0.03	0.10	0.12	0.16	1.23m
	2	1.06	0.04	0.06	0.11	0.18	0.23	1.29m
	3	1.11	0.00	0.04	0.06	0.16	0.27	1.38m
230°F	1	1.13	0.01	0.02	0.08	0.15	0.23	1.36m
	2	1.10	0.00	0.05	0.10	0.18	0.25	1.35m
	3	1.09	0.02	0.03	0.05	0.10	0.13	1.22m
240°F	1	1.09	0.00	0.03	0.07	0.12	0.17	1.26m
	2	1.09	0.01	0.04	0.07	0.14	0.19	1.28m
	3	1.07	0.02	0.05	0.06	0.09	0.17	1.24c

a: adhesive failure mode at the metal interface with the primer
c: cohesive failure mode within the adhesive layer
m: mixed failure mode with some specimens exhibiting interfacial failure and others having cohesive failure within the adhesive layer

APPENDIX D

AFRL/MLSA GRIT-BLAST/SILANE (GBS) PROCESS

Materials

- Solvent (MEK or acetone)
- Rubber Gloves (to conform to applicable safety regulations)
- Dust Mask (to conform to applicable safety regulations)
- Goggles (to conform to applicable safety regulations)
- Respirator (to conform to applicable safety regulations)
- Duralace 9404 Aerospace Wipes (NSN 7920-01-180-0557)
- Nitrogen (dry and oil-free) or verified clean, dry air
- Scotch-Brite, 3M Company (Roloc discs for high-speed grinder; fine grade)
- Aluminum Oxide Grit (50 micron, white)
- Tape (high temperature, such as Teflon, and aluminum-backed)
- Silane Coupling Agent (γ-glicidoxypropyltrimethoxysilane: Dow Corning Z-6040 or equal)
- Distilled or deionized water (pH range 4-6)
- Glass Beaker/Flask
- Graduated Cylinder (to measure out distilled water)
- Pipette (to measure out silane)
- Natural Bristle Brush (such as camel's hair) or acid brush
- BR 127 Adhesive Primer (Cytec Engineered Materials, Inc.)

Equipment

- High-speed Grinder
- Magnetic Mixer and Magnet
- Grit Blaster
- Adhesive Primer Spray Gun
- Infrared Heat Lamps
- Thermocouples
- Hot Bonder or Thermocouple Reader
- Isoscope (or equivalent) or Color Standards to Measure Primer Thickness

1. Solvent wipe an area extending a minimum of 2 feet in all directions around the repair area with methyl ethyl ketone (MEK) or acetone. Choose a solvent that is approved at the repair location. Continue until no residue appears on a clean Duralace wipe.

CAUTION

Appropriate rubber gloves & eye protection must be worn during this operation. An approved respirator may be needed, depending on the application.

CAUTION

Aircraft must be grounded and properly prepared for maintenance per applicable procedures. Fuel tanks must be purged or inerted as required.

2. Remove all organic surface coatings down to bare metal using locally approved procedures. Remove coatings in an area that exceeds the patch dimensions in all directions by at least one inch and preferably two inches.

NOTE

The area within which the surface coatings are removed must be minimized in order to maintain as much of the existing corrosion protection scheme as possible.

3. Solvent wipe the repair area (at least 4 inches in all directions beyond the edge of the repair) using Duralace wipes moistened with reagent grade MEK or acetone. Wipe until all residue is removed (new Duralace wipes remain clean after wiping). Be careful not to drag contaminants into the repair area from the surrounding structure. On the last wipe, remove the solvent with a second clean wipe prior to its evaporation.

4. Connect high-speed grinder to 90 psi oil-free nitrogen (or verified clean, dry air) and abrade the repair area using a new red (fine grit) Scotch-Brite Roloc disc.

CAUTION

Appropriate rubber gloves, dust mask, and eye protection must be worn during this operation. An approved respirator may be needed, depending on local regulations.

NOTE

> *Only verified oil-free, water-pumped nitrogen(or clean, dry air) is to be used in this entire repair process or contamination of bond surface could result (specific new nozzles, hoses, etc. are only to be dedicated for use with the clean nitrogen or air).*

5. Solvent wipe repair area per the directions in Step 3.

CAUTION

> *Appropriate rubber gloves & eye protection must be worn during this operation. An approved respirator may be needed, depending on the application and/or local regulations.*

NOTE

> *From this point forward, extreme care must be taken to ensure that the repair area is not touched by anything, including fingers, except as outlined below.*

WARNING

> *From this point, the repair process cannot be stopped until, as a minimum, the primer is cured. Preferably, the process will not be stopped prior to adhesive curing.*

6. In a clean glass or polyethylene beaker or flask, mix a solution of 150 parts (by volume) of distilled or deionized water and 2 parts of silane; this hydrolyzes the silane. Each 150ml volume of solution can easily treat a square foot (12in x 12in) repair area. This step may be performed at any time as long as the time restraints of Step 7 are met. The water must have a pH between 4 and 6.

7. Place a clean mixing magnet in the beaker, cover to keep from contamination, and agitate on a magnetic mixer for a minimum of 1 hour prior to use. Continue agitating until the solution is actually used.

NOTE

> *Maximum solution life for best performance is 1 hour after the initial 1-hour mix time. Four hours after mix time is the maximum solution life.*

8. Install a grit containment arrangement around the perimeter of the abraded area. It is important (critical for some applications) that the grit be completely contained so it cannot contaminate aircraft systems. The containment should be airtight at the seams and against the aircraft structure. A vacuum cleaner, with appropriate filter for 50-micron aluminum oxide, is necessary to remove grit from the containment unit during blasting in order to maintain adequate visibility. An acrylic box with airtight ports for the blaster, the workers' hands, and the vacuum cleaner hose makes a good containment setup (an on-aircraft "glovebox"). A port with a filter is required to allow air into the box during vacuum cleaner operation.

9. Grit blast an area one inch in all directions greater than the repair with 50-micron aluminum oxide grit using 30-100 psi oil-free nitrogen (or clean air) pressure. Slightly overlap with each pass across the surface.

CAUTION

Appropriate rubber gloves must be worn during this operation. Eye protection and a dust mask must also be worn. If the grit containment arrangement is not airtight, an approved respirator may be needed, depending on local regulations.

Fabrication of grit blast containment box

Use the following guidelines (Figure D-1) to construct a grit containment box:

- Use aluminum or acrylic sheet; windows are necessary to view the working area.
- Incorporate adjustable vacuum feet to allow positioning over the repair area on the aircraft.
- Provide a port for the blast nozzle.
- Provide ports for working gloves.
- Provide a port for a shop vacuum with HEPA filter to collect the used grit.
- Incorporate a filtered port to allow air flow into the container during shop vacuum use.
- Seal the joints between the box and the aircraft structure with tape to prevent grit from escaping.

Figure D-1: Grit Containment Box

NOTE

A fine, matte finish is required. This is a light grit blast - do not dwell in any one area. Ideal blast pressure is dependent on the angle of the nozzle to the surface and the speed at which the blaster traverses over the surface. An angle of 45° to the surface presents a less narrow blast stream; this is helpful in obtaining a uniformly blasted surface, especially if small, circular nozzles are used. Each operator must find the combination of blasting parameters, which leads to a uniform, matte finish.

10. Remove as much excess grit from the repair area as possible by blowing off with dry, oil-free nitrogen (or air), then remove the grit containment arrangement. All grit will not be removed from the surface.

CAUTION

Appropriate rubber gloves must be worn during this operation. If the grit containment arrangement is not airtight, eye protection and a dust mask must also be worn. An approved respirator may be needed, depending on local regulations.

11. Rinse a clean natural bristle brush with distilled or deionized water and use it to apply the silane solution to the repair area for a minimum of 10 minutes after the repair area is wet. First, apply a thin film of the solution to the entire repair area and check for water breaks. Continue to apply silane solution to the repair area to maintain a film of solution on the surface throughout the 10-minute period. Brush the silane from the center toward the edges of the repair area. Do not allow the surface to dry. The solution will pick up grit from the surface as the brush is continually dipped into the mixture.

WARNING

If a water-break-free surface is not achieved, the process must be restarted at step 3.

NOTE

Ensure that the brush does not touch the surrounding uncleaned structure.

CAUTION

Appropriate rubber gloves & eye protection must be worn during this operation.

12. Connect an air nozzle to dry and oil-free nitrogen or air (about 30 psi) and remove the silane solution from the repair area by starting at the center of the repair area and working outward in all directions. A small amount of streaking may be noticeable on the surface after the silane is blown off and the surface is dry.

CAUTION

Appropriate rubber gloves & eye protection must be worn during this operation.

NOTE

Do not allow anything to be blown onto the silane-treated surface from the surrounding areas.

NOTE

The recommended time allowed between completion of silane application and application of heat is 30 minutes. In no case allow this time to exceed 60 minutes.

13. Place a minimum of 8 thermocouples around the perimeter of the silane-treated area but not touching the area to be bonded. Thermocouples must be placed over the various types of substructure that are present (soft skin, stiffeners, etc.) since these will each conduct heat differently. Their placement should be tied to a previous thermal survey of the area. The thermocouple ends must be in contact with the skin and must be taped with high temperature tape (such as Teflon tape). The thermocouples must also be insulated so they are reading the surface temperature and are not directly heated by the heat lamps, such as by covering them with aluminum-backed tape. A recording of the thermocouple locations must be made so meaningful adjustments to the power settings or repositioning of the lamps can be accomplished after heating starts in order to get the optimal temperature spread across the repair area. Plug thermocouples into hot bonder or thermocouple reading device.

14. Insulate substructure, if required. Thermal surveys (on the actual part or an identical part) should have been conducted in order to ascertain that the heating method(s) selected, coupled with the proper insulation, will produce the desired temperatures and tolerances.

15. Center the heat lamps over the repair area. The lamp elements themselves are typically some distance from the surface (for example, 6-12 inches) in order to effect proper heating of the repair area without overheating. If the lamp set-up has a shroud, it should be within about ¼ inch of the structure.

NOTE

A heat lamp set-up containing several independently controlled lamps within a shroud has proven to be effective for many applications. Independent lamp control is important for repair areas that heat unevenly. The shroud helps produce uniform heating and minimizes airflow over the repair area. Thermal surveys must be used to develop the heating arrangement for a given application.

WARNING

Be sure the heat lamp set-up is electrically insulated from the aircraft structure.

16. Plug heat lamps into the power supply and apply heat to the repair area, raising the highest thermocouple reading to 225°F to 230°F. Heat-up rate can be rapid (there is no upper limit on heat-up rate). Do not allow the temperature to exceed 230°F on the repair surface. When all thermocouples are in the range of 210°F to 230°F, maintain heat for 60+5/-0 minutes.

The power setting(s) will need to be adjusted to ensure thermocouples remain in the desired temperature range; monitor the thermocouples and adjust the power setting(s) as necessary. Record temperatures throughout the cure cycle at 10-minute intervals. Prevent cross ventilation to the repair area as much as possible. If necessary, a temperature range of 200°F to 230°F (or even 240°F) may be used.

17. After the 60-minute silane heat dry, turn off power supply, remove heat lamp set-up, and allow the repair area to cool to a maximum temperature of 90°F.

18. Connect primer spray gun to dry, oil-free nitrogen (or clean, dry air source) and spray BR 127 adhesive primer onto the repair area to obtain a cured film thickness of 0.0001 to 0.0003 inches (0.1 to 0.3 mil).

CAUTION

Appropriate rubber gloves, eye protection, and an approved respirator must be worn during this operation.

WARNING

The primer must be warmed to ambient temperature prior to opening the container. A good indicator of this is when moisture no longer condenses on the can.

WARNING

Primer thickness is critical. If too thin, the primer will not cure. If too thick, bond strength will be severely degraded.

19. Allow primer to dry at ambient conditions for a minimum of 30 minutes.

20. Center heat lamps over the repair area and bring it to the predetermined distance from the structure (determined via thermal survey).

21. Plug heat lamps into the power supply and apply heat the repair area, raising the lowest thermocouple reading above 200°F. Heat-up rate can be quick (10-15 degrees per minute if the primer is sufficiently dried at ambient to remove the solvents). Do not allow the temperature to exceed 260°F on the repair surface (lower if dictated by the particular

structure being repaired). When all thermocouples read 200°F or higher, maintain heat for 60 to 90 minutes, depending on the repair surface temperature, as shown below. The heat lamp power setting(s) will need to be adjusted to ensure thermocouples remain in the desired temperature range; monitor the thermocouples and adjust the power setting(s) as necessary. Record temperatures throughout the cure cycle at 10-minute intervals. Prevent cross ventilation to the repair area as much as possible.

BR 127 Adhesive Primer Cure Schedule

Lowest Thermocouple Reading (°F)	Cure Time (Minutes)
200-239	90+5/-0
240-265	60+5/-0

22. After primer cure, turn off power, remove heat lamp set-up, and allow the repair area to cool to a maximum temperature of 90°F.

23. Check adhesive primer thickness using an Isoscope (or similar eddy current device) properly calibrated for the 0.0001 to 0.0003-inch primer thickness or by the use of color standards. The eddy current devices tend to have some problem producing consistent readings on the grit-blasted surface. This can be resolved by spraying onto an adjacent area, near the repair surface, that has not been grit blasted. Color standards must be made (with verified thicknesses) on a grit-blasted surface or must be compared to an adjacent nonblasted area. Use of the color standards to aid primer application prior to cure can be difficult since the color of the cured primer differs somewhat from the uncured condition.

NOTE

If adhesive primer thickness is outside of the desired range, the process must be restarted at Step 3.

Additional Comments

A. The first two steps in the procedure involve removal of organic coatings and corrosion protection schemes; they are not applicable to preparation of new aluminum in the laboratory.

B. The above procedures are based on heat application by infrared lamps. Any heating method can be used as long as the required temperature ranges can be obtained and held for the required times in a safe manner without touching or contaminating the repair surface.

C. Thermal surveys must be conducted on representative structure in order to be sure the heat lamps (or any other device) can properly heat the repair surface within acceptable tolerances. The distance of the bulbs/elements from the surface is one important variable. Since they will inevitably be some distance from the surface, a shroud is usually required to protect the surface from air flow which would cool the area and provide for inconsistent heating as the air flow varied. A metal shroud attached to the heat lamp must be electrically insulated from the aircraft surface. The distance of the heat elements from the surface is dependent on the particular heat lamp set-up and the thermal survey for the application. The arrangement must allow the required temperature range to be achieved in the area without overheating. Stand-off distnaces of 6-12 inches are common.

D. Grit blasting off-aircraft is best conducted in a glove box. A vacuum cleaner (capable of handling the 50-micron grit) is helpful in removing grit during blasting in order to maintain good visibility during the blasting process.

E. Silane heat dry off-aircraft is best conducted by placing the components in a preheated (220°F±5°F) circulating air oven for 60 minutes. Adhesive primer cure is best conducted in an oven per standard off-aircraft procedures.

F. Brushing with a clean, soft brush may be useful (in conjunction with the clean nitrogen or air) during Step 10 in order to remove the maximum amount of residual grit. Care must be taken not to damage the blasted surface.

G. Personnel performing surface preparations must be properly trained. All process steps must be followed carefully since process control is the primary means to assure proper surface preparation. Otherwise, bond strength and/or bond durability will likely be unacceptable. The ability of the personnel to properly perform the process must be demonstrated prior conducting actual repairs. Grit blasting and priming steps require practice. Capability should be demonstrated by preparing wedge test (ASTM D 3762) panels.

H. It is most convenient to prepackage 1 or 2-ml amounts of silane coupling agent in clean vials. A 1-ml pipette can be used to measure the silane. During the repair process, water can be measured into the mixing beaker/flask using a graduated cylinder, and the appropriate number of prepackaged silane vials can be added to make the 1-2% solution of Step 6.